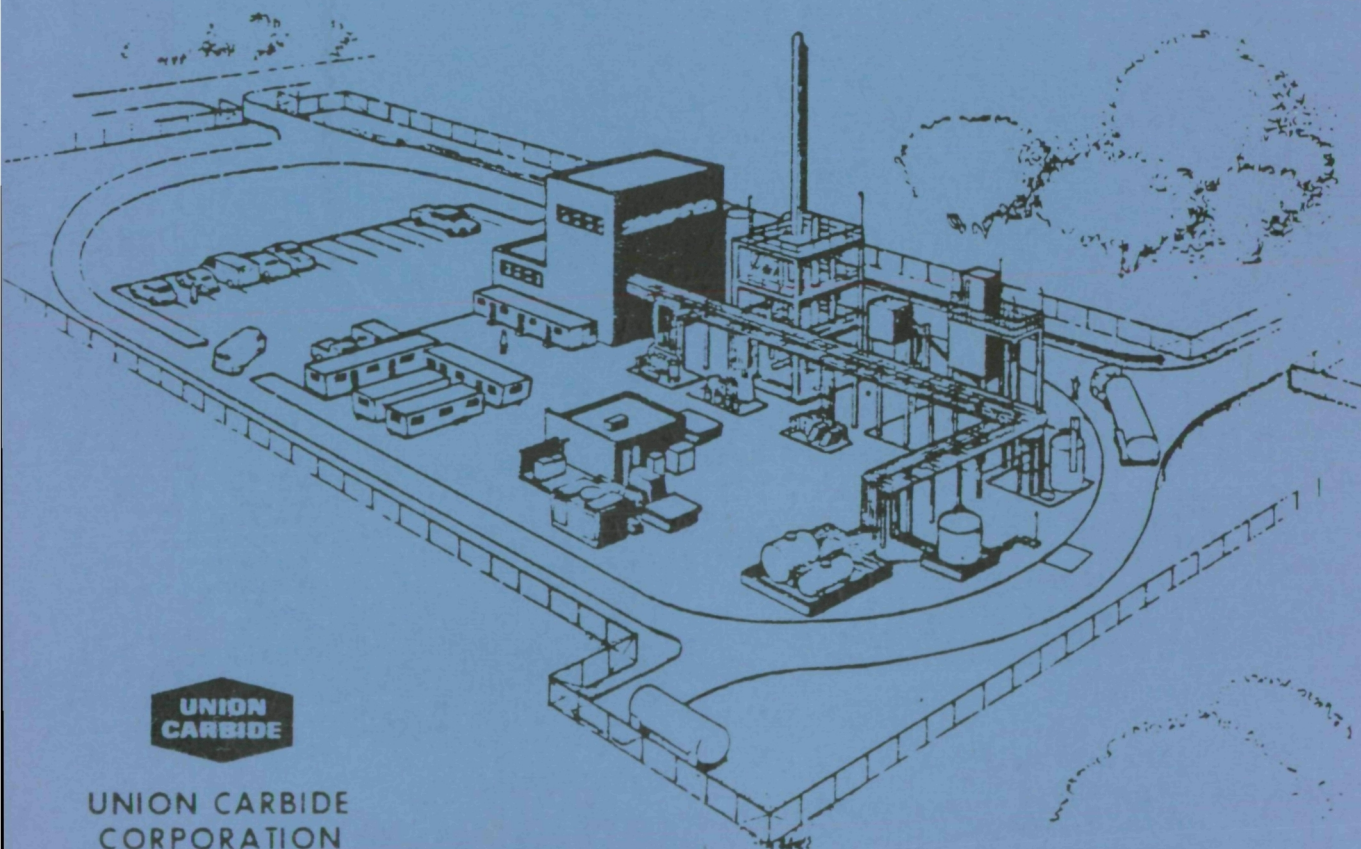FINAL REPORT
PHASE III

APRIL 1979 – DECEMBER 1981

DOE/JPL CONTRACT 954334

Flat-plate Solar Array Project

EXPERIMENTAL PROCESS SYSTEM DEVELOPMENT UNIT FOR
PRODUCING SEMICONDUCTOR-GRADE SILICON USING THE
SILANE-TO-SILICON PROCESS

UNION CARBIDE

UNION CARBIDE
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PHASE III

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EXPERIMENTAL PROCESS SYSTEM DEVELOPMENT UNIT FOR

PRODUCING SEMICONDUCTOR-GRADE SILICON USING THE

SILANE-TO-SILICON PROCESS

FINAL REPORT (PHASE III)

PERIOD COVERED: APRIL 1979 - DECEMBER 1981

JET PROPULSION LABORATORY (JPL) CONTRACT   954334

ABSTRACT

The commercial production of low-cost semiconductor-grade silicon is
an essential requirement of the JPL/DOE (Department of Energy) Flat-Plate*
Solar Array (FSA) project. A 1000-metric-ton-per-year commercial facility
using the Union Carbide Silane Process was estimated to produce molten
silicon for a price of $7.56/kg (1975 dollars, private financing), meeting
the DOE goal of less than $10/kg.

During Phases I and II (refer to June 1979 Final Report), the basic
technology was demonstrated by laboratory experiments that silane can be
produced via a closed-loop chemical process and that silane can be pyrolyzed
in a free-space reactor to produce polycrystalline silicon powder. The ob-
jective of Phase III for the establishment of the practicality of a process
producing semiconductor-grade silicon by a two-step process, the preparation
of silane and the subsequent pyrolysis of silane to yield high-purity silicon,
was (1) the detailed engineering design, fabrication, installation, checkout,
and operation of an Experimental Process System Development Unit (EPSDU),

*Formerly called "Low Cost Solar Array Project"

(2) the performance of a supporting research and development program to provide an information and quality control base usable for the EPSDU and for technological design and economic analyses for potential scale-up of the process, and (3) the performance of iterative economic analyses of the estimated product cost and of large-scale plant cost for production of semiconductor-grade silicon in a EPSDU capable of 100 metric tons of silicon per year.

Because of a severe funding recision in 1981, only a part of the Phase III objective was accomplished, viz:

Task	Description	Status
1.1	Design/Procurement	Completed.
1.2	Equip. Fabrication/Delivery	Completed.
1.3	Installation/Checkout	Site and civil work completed.
1.4	Operation	Preparation of Operating Manual started.
1.5	Commercial Process Economic Analysis	No budget.
1.6	Process Support R&D	Free-space reactor and shotting R&D completed, and stop work order issued in May 1981 on fluid-bed reactor R&D.
1.7	Program Management	As required.

Phase III program will not be completed as planned. However, negotiations are underway between Union Carbide and DOE/JPL for Union Carbide with its own funding to complete construction of the EPSDU.

1.0 INTRODUCTION

This report presents the summary of work performed by Union Carbide Corporation (UCC) for the Jet Propulsion Laboratory under the Phase III continuation of Contract 954334 during the period April 1979 through December 1981. This contract, a part of the JPL/DOE Flat-Plate Solar Array Project, has the ultimate goal to develop the process technology for the manufacture of semiconductor-grade silicon in a large commercial plant by 1986, at a price less than $14 per kilogram of silicon based on 1975 dollars.

BACKGROUND

Union Carbide began producing metallurgical-grade silicon at Niagara Falls, N.Y., in the 1920's and, in the early 1930's, expanded production capacity by constructing a new facility in Alloy, W. Va. Although the silicon business was sold during 1981, the Corporation retains strong ties to this product area.

Because of a continuing interest in silicon chemistry, a series of research projects that began at the Tonawanda Laboratories in 1940 resulted in the development of several commercially useful silicon compounds. Two processes researched then have direct application to the present silane-to-silicon process viz: the copper catalyzed, high temperature hydrogenation reaction for the production of trichlorosilane and the subsequent technique of redistributing trichlorosilane to dichlorosilane.

These research projects ultimately led to the construction of a large commercial facility for producing silicon compounds in Sistersville, W. Va., in 1955 and to the formation of the Silicones Division in 1956. Further research in many areas over the ensuing years has produced a number of relevant accomplishments:

- Research work at Sistersville in the early 1960's resulted in the perfection of purification processes capable of <u>reducing boron and phosphorous contaminants in trichlorosilane to extremely low levels, and in the development of analytical techniques to detect fractional</u>

parts-per-billion concentrations of these contaminants in trichloro-
silane.

- In the early 1970's chlorosilane redistribution equilibria and ki-
  netics were studied extensively using various catalysts. Rohm and
  Haas' Amberlyst ® A-21 catalyst was found to support highly satis-
  factory redistribution reaction rates for chlorosilanes. This cat-
  alyst was used to produce commercial dichlorosilane by redistributing
  trichlorosilane, and to produce silane by redistributing dichloro-
  silane. These two distribution reactions over A-21 catalyst are used
  in the present silane-silicon process.

- In 1957 the technology for producing semiconductor-grade silicon by
  pyrolyzing trichlorosilane in heated quartz tubes was developed at
  the Parma, Ohio, Research Laboratories, and silane produced by the
  molten-salt process was pyrolyzed to yield silicon metal. The tri-
  chlorosilane pyrolysis technology was used between 1958 and 1963 to
  produce lump semiconductor-grade silicon for the electronics industry.

Although Union Carbide had been heavily involved in many facets of the
silicon and silicones business over the years, the technology was scattered
throughout several divisions and locations. In 1974, a corporate task force
was established to assess Union Carbide's total capability and direct future
activities in a coherent fashion. As a result, the wide expertise was made
available to support a more focused effort. The available technologies in-
cluded silicon chemistry; gas-phase pyrolysis; fluid-bed technology; analy-
tical and electronic evaluation; economic analysis and forecasting; safety,
hazards, and toxicity; molten-salt technology; and process engineering.

Corporate research activities were resumed in early 1975 with a program
for producing high-purity, granular silicon metal by pyrolyzing silane.
Later that year, this process and Union Carbide's background technology were
described in a presentation to JPL. As a result, JPL issued a request for
proposal to study the feasibility of the silane-silicon process for producing
polycrystalline silicon in accordance with the stringent economic goals of

the LSA project. The silane process work was continued under corporate funding until Contract 954334 was awarded by JPL in late 1975.

The work conducted during Phases I and II was documented in a final report, dated June 1979. The Phase III effort which covers the period April 1979 through December 1981 is the subject of this report.

SCOPE OF WORK SUMMARY PERFORMED DURING PHASE III

The planned activity for Phase III was to design, fabricate and operate an Experimental Process System Development Unit (EPSDU) to produce semi-conductor-grade silicon using the silane-to-silicon process. The facility was designed, equipment pressures and installation was started at a site in East Chicago adjacent to a Linde Gas Plant. During 1981, it became apparent that funding would be severely restricted and the program would never be completed without an alternate source of funding.

The process support R&D was carried out in parallel with the EPSDU design and construction activities. The free-space reactor PDU successfully pyrolyzed silane and produced silicon powder and hydrogen. The shotter developed by Kayex used this powder, melted it and produced some polycrystalline silicon shot; unresolved technical problems remained. An alternate method of silane pyrolysis in a fluid bed was also under development. Early test results appeared very promising, but the program was stopped in May 1981 due to funding recision.

EPSDU PROGRAM

As illustrated in Table I, the Phase III program consisted of seven primary (WBS level 2) divisions of effort:

- EPSDU Design and Procurement
- EPSDU Equipment Fabrication and Delivery
- EPSDU Installation and Checkout
- EPSDU Operation
- Commercial (1000 MT/Yr) Process Economic Analysis

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- Program Management

Collectively, these activities encompassed all effort required to attain the program objectives. The subdivisions (WBS levels 3, 4, and 5) define the individual work items that must be performed. The progress for this phase is reported at the work-item level.

1.1   EPSDU DESIGN/PROCUREMENT

During Phases I and II of the program which ended in March 1979, Union Carbide's Silane-to-Silicon process design was essentially completed. Based on this design, the EPSDU engineering effort was initiated under Phase III on June 1, 1979.

The Phase III effort included all engineering, design, and procurement activities necessary to transform the process design into a complete installation-drawing package for EPSDU. The major tasks included process design updates, facility design, equipment design and procurement, installation design, and cost estimating support.

1.1.1   PROCESS DESIGN UPDATE

A process design package was issued on June 1, 1979 and served as a starting point for the detailed design of the EPSDU. The design package contained the heat and mass balance, process description, stream catalog/process flow diagram, and functional specifications for process equipment. The process design package was updated June 2, 1980 to reflect changes due to input from engineering design.

1.1.1.1   Silane Process Design

A process design package was issued in Phase II of this contract and was summarized in the final report for Phases I and II which was issued in June 1979. The process design package was reviewed during the Phase III of this contract and a final process design package was reviewed during the

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Phase III of this contract and a final process design package was issued in June 1980 which contained refinements which have potential for lowering cost, reducing requirements for specialized equipment or alleviating problems during operation. The following refinements are reported as follows:

Silicon Powder Line Size

The line size required for transporting the silicon powder was determined. Silicon powder, produced in the free-space reactor, is expected to be transported in dense phase using hydrogen produced by silane decomposition. Laboratory tests were examined to determine the conveying rates and line sizes required for EPSDU. A solids flow rate of 20 lb/min of silicon conveyed in a 1-inch line appeared to be the most workable. The conveying-gas requirement would be a 6 cfm of air; the corresponding pressure loss is 0.3 psi/100 ft in vertical lines and somewhat less in horizontal lines.

M-G Silicon Storing and Conveying

The handling of M-G silicon from the storage point to the feed port in the hydrogenation reactor was changed to simplify material handling and eliminate a process control problem. In the previous handling system, M-G silicon was gravity-fed from the storage bin to a mechanical blender. After the copper catalyst was added manually and the air atmosphere was changed to nitrogen, the M-G silicon/copper catalyst mixture was gravity fed to a lock hopper. It was then pressurized to 500 psig with hydrogen and pneumatically fed to the hydrogenation reactor using recycled pyrolysis hydrogen.

In the original system, control of the pyrolysis hydrogen to hydrogenation presented a problem. If the hydrogenation reactor pressure make-up demand was low, flow of pyrolysis hydrogen would be too small to convey the M-G silicon/copper catalyst mixture. The problem could have been solved with a more complex control system; however, this solution is not desirable. There was also concern about the possible presence of hydrogen

in the enclosed space on the underside of the M-G silicon storage bin, and about the availability of small size pneumatic conveyor fittings.

A pneumatic conveyor system was evaluated for moving the M-G silicon feed for the hydrogenation reactor from the storage bin to the lock hopper to ascertain if the required size was commercially available. Commercial dense-phase pneumatic conveying systems are larger than those required for EPSDU and would not be cost-effective. Flexible-screw conveyor systems are available which are ideal for this application. This type of conveyor has a bearing housing at the motor-end only; therefore, the conveying spiral, made of spring steel in a non-rigid plastic tube, can be cut to size. The manufacturer has demonstrated the capability to transport similar materials successfully at any angle.

The conveying system was revised by substituting a fluidized blending tank for the mechanical blender, by substituting a tubular conveyor for the pneumatic transfer line, and by rearranging the vessels to allow the M-G silicon/copper catalyst mixture to be gravity-fed to the side of the hydrogenation reactor. The pyrolysis hydrogen, previously used for silicon conveying, was re-routed to the superheated hydrogen feed to the hydrogenation reactor. These revisions solved the process control problem.

Hydrogenation Reactor

The hydrogenation reactor design had been considered overly conservative from a size standpoint; however, there had been no supporting data to justify reducing the size. Relevant data became available from the results of JPL-sponsored work at MIT. The reactor size was reduced to a 30-inch I.D. based on this data. This resizing reduced the reactor cost. The size reduction also permitted acquiring reactor operating data that is more accurate because the kinetics will be spread over more of the reactor length instead of being concentrated at the bottom. Thus, the reaction rate can be calculated more accurately.

Other changes were made to simplify fabrication and preclude potential maintenance problems. The previous reactor configuration had a

distribution cone at the bottom and the inlets for the gas and solids passed through the cone. The initial cost of this assembly was appreciable and the flanging and mechanical provisions necessary for removing the cone for inspection or replacement added other significant costs. The feed parts for the gas and solids were changed. The solid inlet was moved to the lower sidewall, and a thermally-jacketed "ram" valve was incorporated as a start-stop flow control with the ability to clear plugs. The gas inlet was changed to two 6-inch flanged assemblies. Each assembly contains a 2-inch STC and a 2-inch H_2 gas distributor fabricated from perforated pipe. This arrangement keeps the superheated hydrogen separate from the superheated STC to preclude premature reactions that could form extremely corrosive mixtures. These changes mechanically simplify the reactor and provide for easier maintenance and more flexibility for injecting gas into the fluid bed. The flanges also serve as inspection ports for checking the integrity of the reactor shell.

Silane Column

The initial silane column was a 12-inch diameter trayed design with a gravity-returned reflux from a condenser that was located at the same height as the top of the DCS column (70 to 75 feet). The flow was so small that the trays would have had to be welded to the shell to prevent significant bypassing due to manufacturing tolerances. Furthermore, the column would have been built in 15 to 20 sections and welded together. Because of the large amount of welding and the difficulty in cleaning after manufacture, the potential for contamination was substantial. To preclude this problem, the design was changed to a packed column employing protruded metal packing and conventional internals. This design minimizes welding, allows for thorough cleaning after fabrication, and permits disassembly of the column in the field if recleaning is necessary. Because the packed column is simpler and is substantially smaller, there is a concomitant reduction in structural support which results in cost savings.

Dryout System

The EPSDU equipment and piping that will contain chlorosilanes must be dried because moisture reacts with chlorosilanes to produce HCl and

silica gel. The original concept for removing moisture prior to start-up was to run the therminol-driven heaters and the gas superheater on recirculated nitrogen to evaporate moisture prior to introducing chlorosilanes. After the physical size and spatial relationships of equipment were determined, the original drying procedure appeared to be less viable.

Since start-up time is very valuable, a centrally located heater will be used so that drying can be accomplished in one week or less. The cost incurred by adding this unit will be returned in operating time saved. The heater is an electrically driven, 200,000 Btu/Hr, unit supplying 730 SCFM of 250-300°F nitrogen. The heater will be mounted at the base of the gantry with a 3-inch header going up to the first platform. Four take-off valves at grade level, two at the first platform, and four at the top level will be provided. Also, one of the pipes from the gantry to the STC or TCS tanks will be insulated and two vent valves added near the waste treatment area.

Hoses, valves, and fittings are required to make jumpers for dry-out. A portable high-pressure nitrogen cylinder with the necessary fittings will be used to purge through the relief valves. Fifteen new vent valves, two block valves, and different sizes for a few existing vents have been identified on the P&I diagram. Since dryout will be performed after the final leak test, valves have been added so that flanges will not have to be cracked. Also, drain valves will be installed at low spots in the piping and equipment.

Waste Treatment System

The initial waste treatment system employed a series of four burners to hydrolyze/oxidize chlorosilanes and silane. The reaction products were drawn into a system of filters and scrubbers by a common vacuum source. The system recovered silica and hydrogen chloride separately as marketable products and produced a minimum of waste sludges. This system is still considered the best design for a commercial system but it is too expensive to use in a facility with a 2-year life. In keeping with the experimental nature of EPSDU, a significant cost saving can be realized if the hydrogen chloride is not recovered.

The waste treatment process was changed so that the muriatic acid byproduct is now neutralized with caustic soda and disposed of in the sewer as a dilute solution, instead of being concentrated to a nominal 20% acid. Other changes include using a smaller waste gas burner, using a silane recycle instead of disposal, reducing the size of the gas cleaning equipment, removing the second silica bag filter (since clear muriatic is not required), and using once-through acid scrubbers since the acid level does not need to be concentrated. The modified system is significantly lower in cost and is simpler to operate because it contains less equipment. Acid neutralization is an added cost but, by combining this neutralization with that required for other facility wastewater, the impact is minimal. An estimated 30 to 50 gpm of wastewater will come from the acid gas scrubber and up to 50 gpm could be generated from curbed and other areas where process spills, if they occur, will be isolated. The neutralizing tank will be designed to process up to 100 gpm of wastewater. The effluent will have a pH of 6 to 9 and will flow to the municipal sewer.

Spill Collection

The initial spill collection system used a network of concrete troughs with open grating. This presented two problems. A potentially flammable, fuming chlorosilane spill would not be kept localized but could stretch 50 - 75 feet along the trough. The unburied troughs would freeze in the winter unless a significant quantity of water was used for sluicing. Both problems were solved by using a spill collection plan employing localized curbing of potential spill areas and a means of reacting the spill at a controlled rate. This has been accomplished by replacing some deluge water with high-expansion foam and by using sloped pads with an outlet valve near a drain hub. In this way a layer of foam can be spread over the spill to prevent fire and acid fumes, and a firehose can be used at the drain hub for sluicing/reacting the chlorosilane liquids drained from the curbed pad through the outlet valves. This spill disposal technique allows the resulting reaction to proceed at a rate such that the water can absorb the reaction acidity and also effectively sluice the solids to a conventional PVC sewer line. This sewer line runs underground, below frost level, to the central neutrali-

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zation point where the acid water can be neutralized to an acceptable level. Because the hydrolysis of the chlorosilane spill is controlled, the solids formed are of a manageable size and should not plug the sewer.

Instrumentation

Only those indicating controllers at the main panel that are necessary to understand the total plant operation will be located at the main control panel; all others will be installed locally.

Electrical Power Supply

The source of primary electrical power was changed from Linde Specialty Gases to Northern Indiana Public Service Co. (NIPSCO). If Linde Specialty Gases supplies the primary electrical power, EPSDU must provide the power cables and other power feed equipment to the EPSDU site. If NIPSCO supplies the power, they will install the required feed equipment at no cost to EPSDU. The sole motivation for making this change was the cost savings.

Powder Processing

Initially, about 1/4 to 1/3 of the silicon powder produced was to be melted and consolidated; however the design allows all powder to be melted. Although the final process has not been defined, it will probably employ a single pyrolysis reactor and up to three melters. The number of melters will be determined from the results of the melting/consolidation R&D.

Stripper Condenser Receiver

The stripper condenser receiver was eliminated by providing additional gravity head on the stripper condenser and changing the control method from a feed-forward and boil-up based on feed rate to a feed-back and boil-up based on the reflux rate.

Silane Storage Tanks

Silane tank pressure control was simplified by eliminating the individual pressure building coils which directly vaporize silane against the atmosphere, and replacing them with refrigerant-brine temperature modulation. The tank design was further modified by making all connections through the top head. These changes reduced potential leaks and potential silane contamination, and improve safety by facilitating improved tank fireproofing.

Chlorosilane Pumps

The chlorosilane pumps were changed to share spares and use centrifugal pumps when possible. This will permit obtaining experience in the type of pump that must be used in a commercial plant. The diaphragm pumps originally specified for EPSDU are not practical or cost-effective for a commercial plant.

Other Minor Changes

The non-combustible gas vent filter was eliminated by combining its duty with the MG Silicon unloading filter. This reduces the cost and simplifies the operation.

The silane superheater was eliminated because pyrolysis R&D has not demonstrated an advantage for preheating silane before injection into the pyrolysis reactor.

The silicon flaker was eliminated. The bin, which previously received the silicon powder to be flaked, has been retained for sample collection and direct removal of excess powder without densification.

1.1.1.2   Engineering Design Support

Chlorosilane Spills

In support of plant layout activities, a methodology for spill and fire control was developed in conjunction with environmental and safety consultants. The major concern was to provide proper drainage, water deluge, and fume control for chlorosilane spills. Data supplied by Southwest Research Institute, under an industry-wide contract, and consultation with the fire safety engineer at the UCC Sistersville plant indicated that hydrochloric-acid fumes and fire are both major hazards associated with chlorosilane spills.

Virtually all EPSDU process streams are flammable due to the presence of hydrochlorosilanes. The most effective fume-control agent appears to be a high-expansion water-based foam which suppresses fire and cools adjacent equipment. Hydrochlorosilane fires cannot always be extinguished; they must usually be allowed to burn out. Based on this, the chlorosilane tankage and process areas will be diked or curbed to contain spills. A portable high-expansion foam generator will be provided to blanket large spills using water from strategically located hydrants. Small spills will be flushed with water only.

Sluice valves in the contaminant areas can be regulated to allow a controlled discharge of diked liquid to the process sewer. Copious quantities of water will be added to the sewerage to hydrolyze the liquids and move the resulting solids. The wastewater will then drain to the central collector where it will be neutralized with caustic soda to adjust pH to permissible levels (6-9). As a back-up fire-control measure, a water-spray deluge system will be used to protect the support steel in the process area. Chlorosilane storage tanks will not have an in-place deluge system, but will be insulated to protect them from external heat. However, hydrant-supplied water will be available if it is needed.

Painting

Specifications were prepared for primer painting and process equipment cleaning. These specification packages included recommendations by UCC experts at Sistersville. The painting specifications are important because the specified primers will reduce the possibility of stress-crack corrosion in stainless equipment, and external corrosion of steel equipment resulting from chlorosilane leakage. Proper process equipment cleaning specifications should enable a rapid, less troublesome startup.

Waste Burner

The waste burner and the silica agglomerator required special process design attention because of their customized design. The EPSDU waste burners were assembled from components purchased from a burner component manufacturer because the waste disposal system requirements cannot be met with available pre-engineered systems. A manufacturer was contacted to establish component availability and process requirements. The preliminary equipment flow schematic shown on Figures 1.1 and 1.2 were developed for preparing the specifications. There are four burners including one spare. Figure 1.1 shows the schematic of a waste gas burner and Figure 1.2 shows that for the waste liquid burner.

Each burner has an independent natural gas/combustion air piping and pilot system, but the combustion air supply and flame supervision system is shared. Switching of flows from the operating waste-gas burner to the spare burner will be accomplished by a manual switch on the flame-supervision control panel.

Gantry Scale Model

Process engineers assisted in building the gantry scale model which proved invaluable for identifying and resolving problems. Its use permitted solving problems "in plastic" instead of "in steel" and its cost will be more than offset by the cost savings of field delays that have been prevented.

FIGURE 1.1 Process Control Schematic
Waste Gas Burner #1

NOTE:
1.) ALL PIPES ARE SCH. 40 (UNLESS OTHERWISE SPECIFIED)
2.) COMB. AIR CONTROL VALVE (13) TO HAVE 12-15 SECOND STROKE TIME
3.) ORFICES (14) AND (25) TO BE INSTALLED (5) PIPE DIAMETERS DOWNSTREAM
AND (3) PIPE DIAMETERS UPSTREAM OF ANY FITTINGS OR VALVES

SYMBOLS & LINE CODE

------ ELECTRICAL SIGNAL
----- PNEUMATIC SIGNAL OR IMPULSE SIGNAL

TO SS - TO WASTE GAS BURNER #3 SELECTOR SWITCH
TO FS - TO FLAME SUPERVISOR

- N/A SCOPE OF SUPPLY

CA = COMBUSTION AIR
NG = NATURAL GAS
AO = FUEL OIL PORT
AA = ATOMIZING AIR PORT
PLT = PILOT PORT
FD = FLAME DETECTOR

COMBUSTION AIR HEADER, TO SHEET #2,3,4.
4"∅ SCH. 40. FLG'D.

NATURAL GAS HEADER, TO SHEET #2,3,4.
1/2"∅ SCH. 40. SCN'D.

TO LIQUID WASTE BURNER, 459-14, SHEET #4

COMBUSTION HEADER TO PROCESS, SHEET #2,3,4.

COMBUSTION AIR, TO WASTE GAS BURNER #3
NATURAL GAS, TO WASTE GAS BURNER #3
FUEL OIL, TO WASTE GAS BURNER #3
ATOMIZING AIR, TO WASTE GAS BURNER #3
459-12, SHEET NO. 3

10"∅ SCH. 20 STEEL W/2" THK. CALCIUM SILICATE
INSULATION + EXPANDED METAL FOR PERSONAL PROTECTION

FIGURE 1.2 Process Control Schematic
Waste Liquid Burner

Equipment Layout

Process design personnel assisted in the mechanical layout design to assure that equipment location, equipment supports, and piping layout are consistent with safety requirements and operability. Even simple things such as the proper location of the STC pump isolation valve require knowledge of the process chemicals, equipment, control method, and operational mode. Consequently, all layout details were scrutinized carefully.

Equipment cleaning requirements were reviewed. The components which could not be cleaned by vendors were cleaned in accordance with Union Carbide's electronic specifications. Instruments and other sensitive items were also cleaned at Tonawanda, but other items such as pipes will be cleaned on-site.

Relay Logic

The relay logic was basically related to process area: hydrogenation, distillation/redistribution, pyrolysis/consolidation, and waste treatment. The first two areas had the most complex relay logic. Automatic shutdowns and relay interlocks were used whenever rapid, programmable action was necessary to avoid injury to personnel, significant equipment damage, or severe process upset.

Insulation

A potential problem concerning insulation of the high temperature lines from the superheater to the hydrogenation reactor was reviewed and resolved by specifying a preformed insulation with a lower thermal conductivity and greater thickness than shown on the original drawings. If the temperature drop is found excessive during checkout and startup, more insulation will be added to the flanges and valves.

Mechanical Engineering/Installation Design

The drawings were reviewed for compatibility with the P&I diagram. The review was conducted line-by-line according to the P&I diagrams

and recommended changes were incorporated in the P&I diagrams, installation design drawings and the scale model. A construction bid package incorporating these changes was released. Any further necessary changes will be resolved as field changes during construction.

Safety

Time was spent thoroughly reviewing the engineering design from operability and safety points of view. The design was examined for adequate provisions for startup, emergency shutdown, and draining of process fluids. Major and minor problems were identified so that costly field changes will be avoided.

Gas Analysis

A set of data for vapor-liquid equilibrium on a 1% diborane in silane system was received. It was analyzed to insure that the silane column reflux controls will have the correct range. This analysis was performed by re-running the distillation simulation program with the activity coefficients that resulted from the data. The number of trays will be constant and the product purity can be compared to the reflux.

1.1.1.3   Burner Tests

An analytical and experimental program was conducted to provide design data for an economically and technically viable waste treatment system for EPSDU. All components of the system were commercial items except for the burners. Tests were conducted to develop burner components that could flame hydrolyze and neutralize chlorosilane liquid and gaseous wastes. A report was written which describes the testing and results. Excerpts from the report are as follows:

Introduction

The waste treatment system for EPSDU, as with any new chemical processing facility, is extremely important. Provision for an environmentally acceptable method of disposing of wastes must be in place and operating before the primary processing systems can be started up. Since EPSDU is an experimental facility, the waste treatment technique has to be effective but does not necessarily have to be economically optimum.

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There are two types of chlorosilane wastes associated with the EPSDU process — vapors and liquids. Several possibilities for disposing of these materials were investigated. These techniques consisted of contracting for wastes removal, neutralization and disposal off-site and on-site treatment. Experiences from chlorosilane disposal at the UCC Keasbey silane plant and information and ideas from consultants and waste-disposal equipment vendors were used to develop the waste treatment system concept for EPSDU.

The planned scheme was based on using fumed silica burners, gas stream particulate removal equipment, and acid-vapor venturi scrubber equipment to convert all wastes to low value byproduct materials. The basic system uses thermal oxidation to process waste materials that range from silicon tetrachloride (heavily contaminated with metal chlorides) to silane and hydrogen gas. This system is based on the successful thermal oxidation system used at Keasbey for waste disposal, on commercial silicon tetrachloride burners used for producing commercial silica filter material, and on commercially practiced bag filter and particulate removal equipment. The burner system is used to convert metals to their oxides, hydrogen to steam, and chlorine atoms to HCl vapor. The byproducts are muriatic acid and dry silica powder. The acid has potential use in the metallurgical industry for pickling and the silica powder in the rubber of roofing industries for filler.

All components of the waste treatment system are available as commercially proven equipment except for the burners which must be developed from available combustion components. Because of the wide range of properties of EPSDU wastes, it appeared that separate burners would be required for each of the following:

- Liquid waste

- Chlorosilane vapors and nitrogen contaminated with chlorosilane vapors

- Hydrogen-rich and silane-rich wastes

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The liquid wastes consist primarily of mixtures of silicon tetra-chloride and trichlorosilane heavily contaminated with metal chlorides in both dissolved and sludge forms. The wastes must be oxidized in a liquid burner equipped with an atomizing spray nozzle. For the oxidation process, the burner must be provided with enough hydrogen atoms to completely react the chlorine in the silicon tetrachloride to HCL vapor. The hydrogen can be provided by methane (natural gas) feed. Excess hydrogen must be provided to permit the complete conversion of the chlorine to HCL vapor to prevent residual chlorosilane in the combusted gas stream from leaving the burner. A critical control parameter is proper flame temperature which is controlled by the amount of excess air that is admitted with the fuel to the burner. The anticipated optimum flame temperature was expected to be between 1600° to 2000°F. The temperature must be high enough for com-plete combustion of the chlorosilane material. The kinetics of thermal oxi-dation was strongly favored by higher temperatures; however, high temperature operation results in the production of extremely fine silica particulates which are difficult to remove in the downstream particulate removal equipment. Thus, the burner should be operated at a temperature as low as possible to yield larger silica particles that are easier to remove, handle, and market. The relevant design data for the waste liquid burner include:

- Waste feedrate range (expected to be 1.5 - 2 times).

- Atomizing air rate vs waste liquid feedrate to obtain optimum flame geometry and stability.

- Optimum fuel vs silicon tetrachloride waste liquid feedrate ratio required to assure complete oxidation of the waste as determined by the analysis of the downstream flue gas stream.

- Best flame temperature operating point (high enough to assure complete oxidation of the bed, yet as low as possible to re-sult in the best particulate silicon morphology).

A nozzle mix burner is also planned for oxidizing the hydrogen-rich and silane-rich waste streams. Design information for this burner is similar to that of the chlorosilane-rich gas burner, except that hydrogen is not re-quired to balance the chlorine.

The overall waste treatment scheme planned for EPSDU is shown in Figure 1.3. (The burner-related components and streams are identified by heavy lines).

Certain burner performance information is necessary for specifying other system components such as the bag filtering system. This requirement and the need for having the waste treatment section totally operational before EPSDU startup mandated the need for an experimental program. The experiments were limited to the burner system and were directed toward providing the minimum data required to permit designing the EPSDU system with confidence.

Waste-Burner Process Development Unit (PDU), Design Basis

The PDU design was based on using readily available components and temporary installations to generate design data for the burners. The apparatus capability was limited and could only accommodate testing of the burner and evaluation of the reaction stoichiometry.

Direct design group experience in designing for and working with chlorosilane waste neutralization and disposal was limited; however, relevant information was gained from a consultant equipment vendor, and operating personnel at Keasbey and Sistersville.

Keasbey has a waste burner for silane and trichlorosilane vapor. It is crude and oversized; however, by using large amounts of natural gas, it operates effectively if not economically. The energy wasted by this system would prohibit its use in a commercial silane-silicon facility.

Sistersville, which produces most of UCC's chlorosilane chemicals, neutralizes the liquid wastes by injecting them into water. In this batch process, chlorosilane "sludge" (liquid) is injected every few hours at a flow of 50 gallons in a 2-minute period. This technique is expensive, difficult to operate, and very unreliable; however, a similar system, using a slow continuous feed, might be used as a temporary backup for the EPSDU liquid burner.

Gaseous Chloro-silanes Burner

Hydrogen & Silane Burner

Spare

Liquid Burner

Liquid Holding Drum

Air Cooled Heat Exchanger

Bag Filter

Hopper

Water

Scrubber #1 & #2

Tailing Column

Vent

Induction Blower

Lime

Neutralizer Tank

Sewer

PDU only

Vent

Filter

HCl Adsorbers

Vacuum Pump
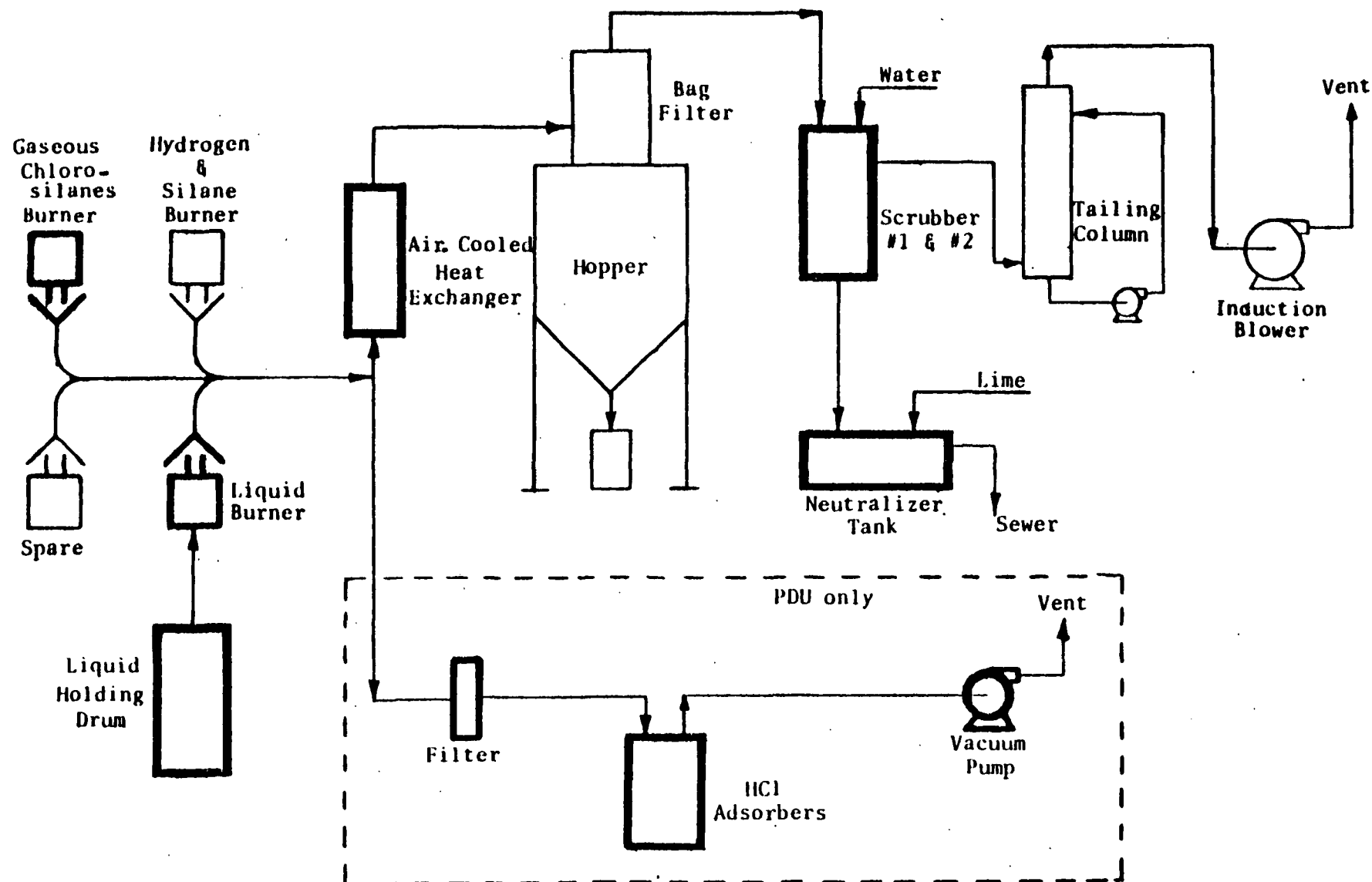
NOTE: MINI-WASTE BURNER PDU BEING TESTED HAS HEAVY LINES

FIGURE 1.3   EPSDU Waste Treatment System

The EPSDU waste treatment system will be on-line at all times and must operate reliably. The waste vapor burners will operate continuously but the waste liquid burner can be operated periodically. The waste chlorosilane vapors must be reacted continuously because storage would be impractical. The waste-gas burner has to be able to process reasonable flows from the plant including emergency situations. The waste-gas burner should also be able to operate from idle to full capacity on demand to prevent silica dust and hydrogen chloride from being released into the atmosphere. The waste-liquid flows from the plan can be more controlled. The liquids can be accumulated in the surge tank so that the burner can be operated for 12 hours and then be taken off-line for 12 hours for cleaning and for repair. These operational modes were a prime consideration for the experimental program.

A commercial air atomizing nozzle was installed. However, it plugged immediately.

Next the sludge was allowed to settle over a weekend without agitation so that runs could be made without solids in the feed. The runs lasted 36 and 34 minutes. It appeared at this point that the problem of reacting (burning) Sistersville TCS sludge was not caused by the solids.

The last test run was made using STC/20% TCS mix with no solids. The burner performed the same as it did with pure STC. The flame was totally within the flame holder and the run, which lasted 1 hour and 8 minutes, produced dust about 1% Cl^-.

Analysis of Test Results

Figure 1.4 shows the variation in Cl^- content of the dust (chronologically) produced during the entire testing period. Note that the STC/TCS mix produced dust of the same quality (about 1% Cl^-) as the STC liquid when the burner operated for 7½ hours. The atomizer nozzle used for the mix was not exactly the same as the one used on STC liquid — the outlet hole was smaller. When the atomizer is made for the EPSDU liquid burner, it should be checked and tested carefully since only small differences in dimensions

26
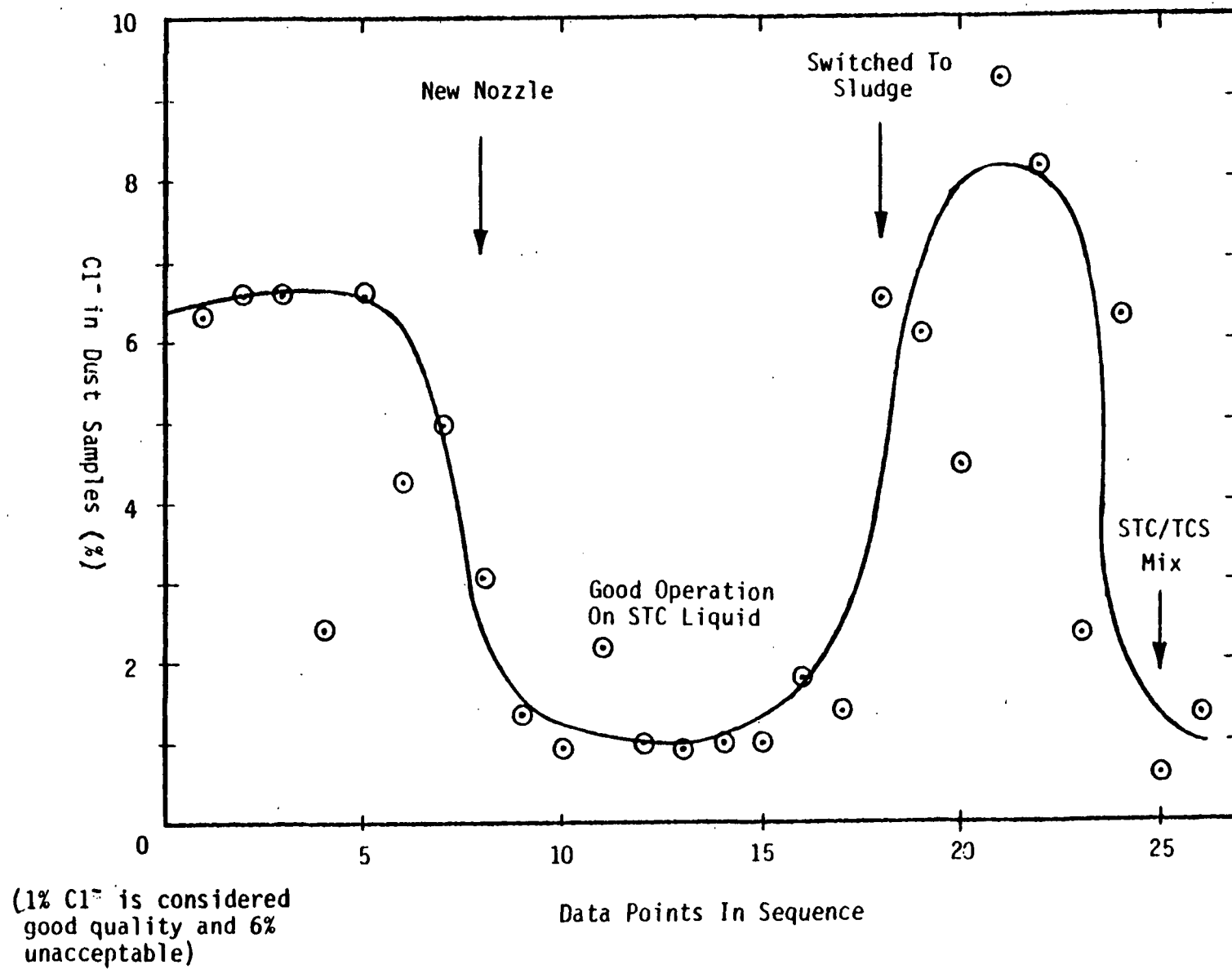
FIGURE 1.4   Quality of Dust Collected During the Waste Burner Test

can significantly impact successful operation. The burner test set-up was photographed and is presented in Figures 1.5 and 1.6.

Table II shows the amount of polysilicons (Si_2Cl_6, Si_3Cl_8, Si_4Cl_{10}) in the Sistersville sludge. It appears that they were causing the problems with the burner. The same type of problems should not occur in the EPSDU.

TABLE II

COMPARISON OF SLUDGE COMPOSITIONS

Sistersville Sludge		Expected EPSDU Sludge
STC	8.7%	81%
TCS	30.9%	16%
Solids	10-15%	3%
Si_2Cl_6	20.5%	
Si_3Cl_8	27.0%	
Si_4Cl_{10}	10.5%	
Heavies	2.3%	

Solids content is an estimate; all other numbers are on a solids free basis.

The build-up in the exhaust piping during the testing was soft and friable for the most part. There was a thin harder layer at the pipe wall. The soft build-up was actually pigged (cleaned) by pulling a piece of paper through the pipe with a string. The harder layer could be easily scraped off with a piece of metal.

At EPSDU, a pig made of spring steel (shown in the sketch) could be turned and pushed forward with a plumber's snake to clean the piping.

PLUMBER'S SNAKE → SPRING STEEL

Exhaust to Scrubber

Flow Control Panel

Burner Controls

Waste Burner

Sample Train

Sample Filter

DANGER HOT

FIGURE 1.5   Waste Burner Test Apparatus

Burner and Receiver

Secondary Air Inlet

Thermocouple

Exhaust to Scrubber
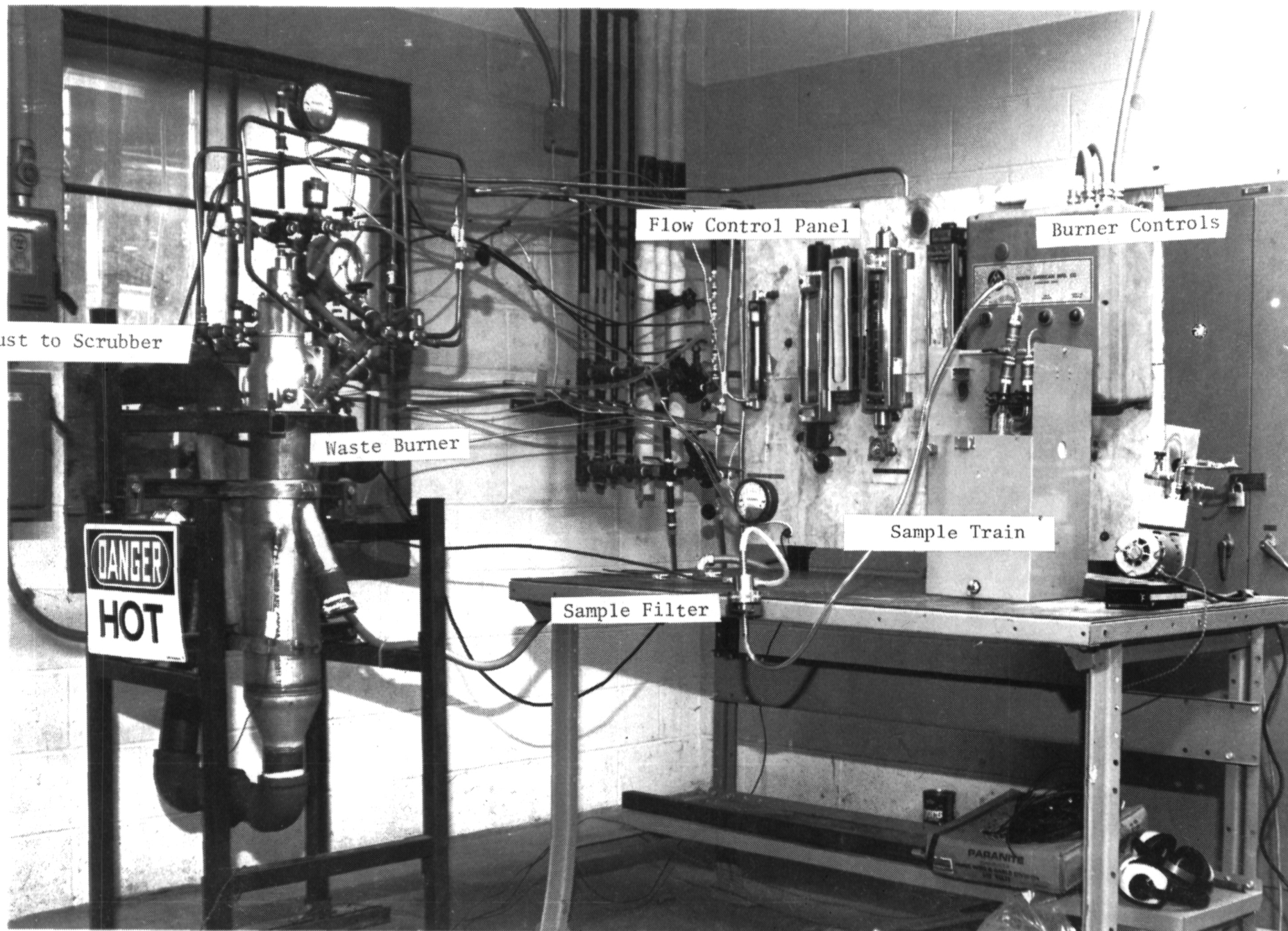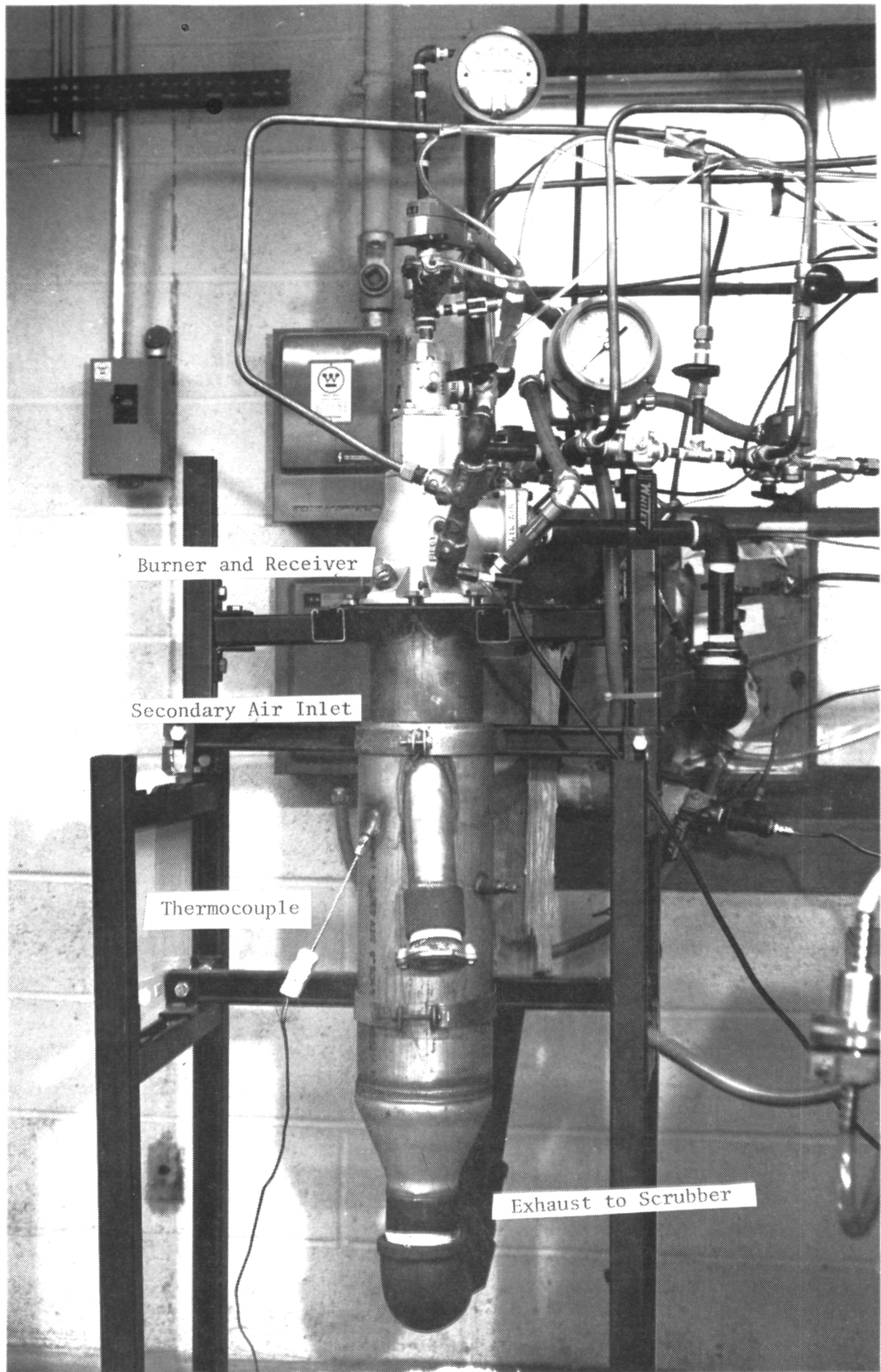
FIGURE 1.6   Waste Burner Assembly

The exhaust piping should be built using long radius welded elbows and the fittings necessary to allow pigging while the system is operating. The exhaust piping should contain sufficient flanges to allow access for cleaning; screwed fittings should be kept to a minimum.

There were about 700 lb of chlorosilane left after the last test run. This was neutralized by injecting it under water and adding lime to neutralize the HCl produced. An injection rate of 50 to 150 lb/hr worked quite well; the injection tube did not plug very often and only a small amount of HCl escaped from the neutralizing tank. The 1/3 hp agitator kept the tank well stirred so only a minimal amount of hand stirring was needed. This scheme would make a reliable back-up system for the liquid burner at EPSDU for short-term emergency use. Injecting the liquid chlorosilane into water is a method that should be considered when neutralization is required. If the process is conducted at a slow continuous rate, it will be adequate to process the average rate of 36 lb/hr projected for EPSDU.

A number of the valves were disassembled after the test. The exteriors of the valves were corroded but the interiors were in good shape except for some hydrolyzed silica that formed after they were disassembled. Most of the elastometers, Buna N, and the tygon tubing had hardened. One piece of Viton tubing was still in excellent shape after only 3 hours of chlorosilane service. During the early part of the test, many problems arose because of the hydrolyzate in the valves and piping. As purging and operating procedures improved, the problems disappeared. Inspection of one check valve showed enough hydrolyzate to make it inoperative. This shows that thorough purging is required prior to introducing chlorosilanes and before disassembly.

A material inspection was performed on the flame holder, flame receiver, and a piece of the cooling pipe. The inspection showed that 304SS is acceptable for the flame holder and flame receiver. Carbon steel will work for the cooling pipe and fittings but cast iron is inadequate. The piping system must be heated prior to putting chlorosilanes into the burner.

Conclusions

The primary purpose for conducting the burner tests was to develop information necessary for designing a reliable waste treatment system for EPSDU. Although the test program was hampered by cold weather, schedule and equipment problems, it was a practical success in that information necessary for designing and operating the burner system with confidence was generated. The system concept should work effectively, but additional development may be required after installation at EPSDU to provide the refinements necessary to ensure reliable operation.

Regarding overall burner system operation, the natural gas to waste chlorosilane ratio had little effect over the range tested and the amount of excess air and flame temperature did not have any gross effects. At higher excess air rates and lower temperatures, the dust particles were larger but contained more chlorine (not completely reacted).

The waste gas burners should not pose any significant problems. During the tests, the chlorosilane/nitrogen mixtures were flame hydrolyzed at low rates with high hydrogen-to-chlorine ratios with satisfactory results. The burner for EPSDU should be sized to allow higher hydrogen-to-chlorine ratios if they become necessary.

The liquid burner at EPSDU may cause some problems, but the system should perform adequately. The main problem is attaining the proper atomization necessary to provide the mist of fine droplets with the low forward velocity required for the reaction. Although a workable nozzle configuration was identified during the test program, it could not be considered a final design because representative EPSDU-type sludge was not available for testing.

Handling chlorosilanes was more difficult than expected. Knowledgable control of the liquid is required for safe, dependable operation. Proper equipment location and installation will assure suitable operation and eliminate hazardous situations. All equipment and piping that comes in contact with chlorosilanes must be thoroughly purged. Valving should be provided to

permit purging of each piece of equipment that may require maintenance during plant operation or after shutdown.

Solids were deposited in the piping during all testing; however, the deposits were light and easily scraped off. Design of some internal solids-removal device will be required. The elbow at the ejector inlet had a different type of build-up than the rest of the exhaust piping. It consisted of a heavy wet mass caused by water back spray from the ejector reacting with chlorine left in the dust. Wet scrubbers used for gas cleaning should have a well-defined water inlet to prevent back spray. At EPSDU, a redesign of the present configuration could result in a system that prevented accumulation areas. If these areas cannot be eliminated entirely, the redesign should provide for a cleaning capability during operation.

Experience from these tests should be used in the selection of compatible instrumentation. All-metal rotameters did not provide the prescribed accuracy but glass tube rotameters responded satisfactorily. The UV detector flame eye worked on the dusty flame only if it was cleaned every two days or if a small air purge was applied. Measurement of the flame temperature with thermocouples provided readings lower than expected.

In the event of an unexpected shutdown of the EPSDU Waste Treatment Area, a standby system should be available. This system should be capable of storing at least 10 days EPSDU waste accumulation. In addition, equipment required to inject the liquid into water (neutralize chlorosilanes), should be purchased and stored at EPSDU. The system of piping, valving, and local instrumentation could be built around temporary storage in appropriate types of 55-gallon drums. After the testing, the leftover chlorosilanes could be disposed of by injecting them under water, adding lime to neutralize the acid, and flushing them down the drain. This process is similar to the process used at Sistersville except a slow controlled rate of 50 to 150 lb/hr was used. With a properly installed reactor vessel and sufficient agitation, wastewater could be flushed to the sewer.

The waste treatment area should be started up at least two months before EPSDU startup. This will enable plant personnel to become familiar

with the system and prevent operational problems that could impact the overall program schedule.

1.1.1.4   Pyrolysis/Consolidation Process Design

Process Design Package

The process design package for the pyrolysis/consolidation section was completed and issued in June, 1981. This design package was used as the basis for developing the P&I diagrams and design in Task 1.1.6 (Pyrolysis/Melting System Design). The text is divided into ten sections viz:

1. Process Description
2. Process Flow Diagram
3. Heat and Mass Balance
4. Equipment List and Functional Specifications
5. Preliminary Layout
6. Utility Requirements
7. Special Requirements
8. Safety Considerations and Information
9. Shotting Tower Drawings
10. Free-Space Reactor Fabrication Design

Process Flow Diagram, Heat and Mass Balance

A process flow diagram was developed to show the interconnections between equipment items. A heat and mass balance analysis was made for the process and a stream catalog developed defining temperature, pressure, flow rate, molecular weight and composition of each fluid.

Process Description

Silane is withdrawn from the top of the Silane Storage Tanks, the contents of which have been analyzed to confirm adequate purity. The pressure and flow controlled silane is injected into the Free-Space Reactor through a water-cooled stainless steel nozzle. The walls of the reactor are

heated to 900°C by the Free-Space Reactor Pyrolysis Furnace. This is an electrical resistance multi-zone furnace which is connected to plant power through a transformer. Energy is transferred convectively from the reactor walls to the entering silane jet via recirculating product hydrogen and silicon. Above 400°C the silane rapidly decomposes according to the overall reaction:

$$SiH_4 \ (g) \rightarrow Si \ (s) + 2H_2 \ (g)$$
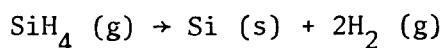
The reaction is essentially complete before the silane reaches the walls of the reactor, thus precluding any hard deposit formation. The product silicon is in the form of a very fine (0.4 μm) powder which is periodically stripped from the reactor wall by a mechanical scraper. The silicon is separated from its hydrogen carrier by porous metal filters located in the lower chamber of the reactor. The pyrolysis hydrogen exits the hopper, is cooled to 100°F in two steps against cooling water (Pyrolysis Hydrogen Precooler, and Pyrolysis Hydrogen Cooler), and enters the first stage of the Pyrolysis Hydrogen Recycle Compressor. A portion of the hydrogen is recycled from the first stage to the Free-Space Reactor and to the shotter assembly. The remainder enters the second stage of the compressor and serves as make-up hydrogen to the hydrogenation reactor.

The silicon powder in the lower reactor is cooled by recycle hydrogen and the cooled powder periodically is dense-phase-conveyed to the Shotter Feed Hopper. Compressed hydrogen stored in the Hydrogen Accumulator maintains hopper pressure during conveying. Following atmosphere exchange with argon, the powder is fed to the shotter via a variable speed screw auger. The powder is then melted and forced through a (nominal) 1 mm orifice to form (nominal) 2 mm shot. The shot solidifies in the shotter drop tube and is cooled to close to ambient by recirculating hydrogen in the Shot Receiver. The hydrogen used is cooled against cooling water in the Shot Receiver Hydrogen Recycle Cooler, filtered, boosted in pressure by the Shot Receiver Hydrogen Recycle Blower and reintroduced into the Receiver. Cooled shot is removed via the Shot Lockhopper and drummed. The collected shot is weighed on the Shot Scale and sent to storage.

Powder produced in excess of the shotter's capability is conveyed
to the Excess Powder Hopper. Following atmosphere exchange with nitrogen,
the powder is conveyed into drums, weighed on a scale and sent to storage.

Equipment

An equipment list was prepared along with functional specifications
for each of 27 equipment items. A preliminary layout of the Pyrolysis/Consol-
idation building was developed which shows the location of equipment, items
related to Q.C., utilities and non-process related features.

Silicon Powder Properties

An analysis of the flow properties of the free-space reactor sil-
icon powder was made to determine design parameters for a transport system.

1.1.2   FACILITY DESIGN

Facility design consisted primarily of the effort required to
translate the process design functional requirements into specific plans re-
garding site, physical arrangement, human factors, and safety and environ-
mental considerations. Personnel who will operate the facility participated
to provide human factor inputs and to become familiar with the process.

1.1.2.I   Layout, Site Evaluation

This effort included the preparation of layout, plot-plan, and
evaluation drawings to establish the spatial relationship of equipment and
structures and provided the design basis for detail piping, structural steel,
foundations, and electrical systems. Site evaluation included definition of
battery limits and utility connections, topographical surveys, and soil
testing. Figure 1.7 shows an artist rendering of the overall facillity.

The following drawings were prepared for use in the Installation design effort:

SKD-127181	Site Plan
SKD-127256	Gantry
SKF-127259	Layout
SKD-131839	Roadway
SKF-131852	Melter Building
SKF-131853	Cooling Tower
SKF-131855	STC and DCS Storage Tanks
SKF-131857	M-G Silicon System
SKF-131858	Silane Shift Tanks
SKD-131873	Proposed Routing & Tie-Ins of Underground Utilities Outside Site
SKD-131882	Underground Utilities Routing - Area A and Area B

The utilities shown in the last two listed drawings include 12.5 KV power and a 3-inch gas line from NIPSCO, a 2-inch nitrogen line and a 4-inch pressurized sewer line from the Linde Specialty Gases facility, and a 6-inch fire water/potable water header connected to the recently-installed Specialty Gases supply loop.

Drawing SKD-131891, Layout of Switchgear Building, also released, included the following equipment:

Main instrument panel
Scanivalve panel
Emergency generator panel
Motor control center #1
Motor control center #2
2500A bus duct
12.5 KV/480 V transformer
Primary disconnect switch
325 KW emergency generator
Emergency generator fuel tank

SILICON for solar power

EXPERIMENTAL UNIT
UNITED STATES DEPARTMENT OF ENERGY
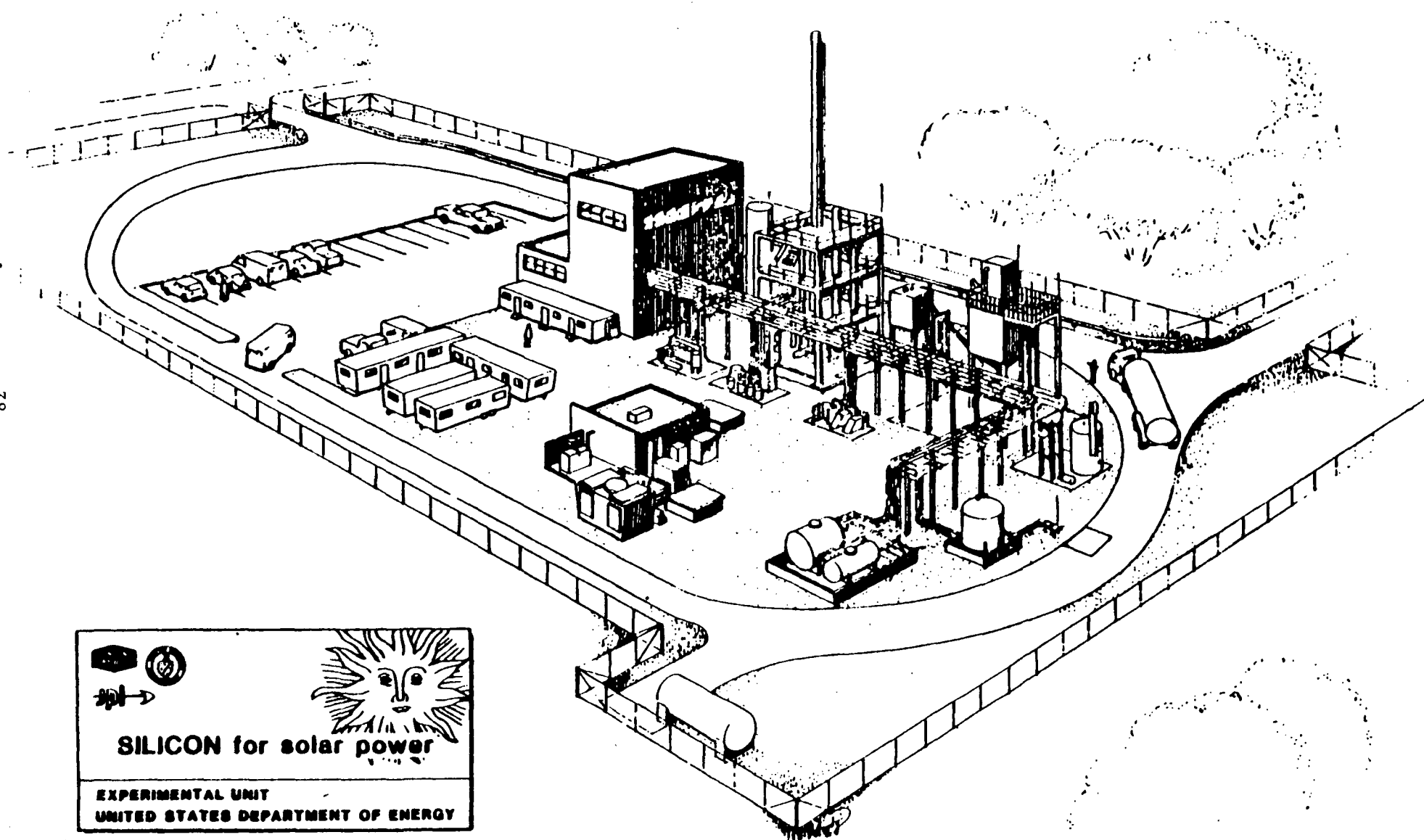
FIGURE 1.7   Artist's Sketch of EPSDU Facility

100 psig/250 cfh instrument air compressor

100 psig/250 cfh instrument air dryer

Within the EPSDU site, the power, natural gas, nitrogen, sewer and water lines are routed to their specific use points. For example, underground fire-water lines supply four fire hydrants around the perimeter of the facility and seven personnel safety showers. Two local natural gas supply lines off the main line will serve the therminol furnace and the superheater furnace. Drain/sewer lines tie the various chlorosilane liquid use points (storage tanks, pumps, gantry, Q.C. trailer) to a common neutralizer — emergency spill basin. Sanitary wastes from personnel trailers are collected in a septic basin prior to transfer into the existing plant system.

A major concern in EPSDU design was the control of hydrochloric acid fumes and fires associated with chlorosilane spills. Virtually all streams are flammable due to the presence of hydrochlorosilanes. A spill and fire control methodology was developed using data supplied by Southwest Research Institute under an industry-wide contract, and consultation with the fire safety engineer at the UCC Sistersville Plant. The major requirement was to provide proper drainage, water deluge, and fume control. The most effective fume control agent is a high-expansion water-based foam which also suppresses fire and cools adjacent equipment. Hydrochlorosilane fires cannot be extinguished but will burn until the fuel is consumed.

Spill and Fire Control Methodology

Based on the selected methodology, a layout was developed for the chlorosilane tankage and process areas. These areas were curbed to contain not less than 1.3 times the volume of the largest tank in the area. A portable high-expansion foam generator is used to blanket large spills using water from strategically-located fire hydrants. Small spills will be flushed with water only. Sluice valves were provided in the containment area to allow a controlled flow of curbed liquid to discharge to the process sewer. Copious quantities of water will be added to hydrolyze the discharge and move the solids. The resulting wastewater will be neutralized with

caustic in an automated pH control system and discharged into the local sewer. To reduce the hazard of delayed hydrogen evolution, short open trenches were provided in the process area and untrapped, vented pipe connected the tank storage area to the catch basin.

A water-spray deluge system was used to protect the gantry steel in the process area. This system can flood the ground level with water at the rate of 0.2 gpm/ft^2 and the upper levels with 0.5 gpm/ft^2. Chlorosilane tanks do not have an in-place deluge system but were insulated to reduce heat input due to fire. Any point in the process area can be covered by at least two fire hoses from different hydrants.

Layout Description

The EPSDU facility is located at the Linde Specialty Gases/Gas Products site at East Chicago, Indiana. Figure 1.8 (Dwg. SKF-127259E) shows the working area of the site. The total site is approximately 485 feet long by 225 feet wide. The working area is encircled by an oval-shaped perimeter road with a counter-clockwise traffic pattern. Entrance and exit is at the southwest corner. An emergency gate, located at the southwest corner, is up-wind of the process area during the prevailing southwest winds. A truck un-loading pad is located on the perimeter road at the west end of the facility near the caustic, chlorosilane, and fuel oil storage tanks. Parking, visitors trailer, and the construction trailer are on the east end of the site. Operating, Maintenance, Engineering, and Locker Room trailers are on the north side of the site near the parking and perimeter road. The major portion of the process area is on the south side of the site including; the process gantry, pyrolysis/melting/consolidation building, and the waste burner area.

Additional safety features incorporated in the layout and equipment design include:

● Silane tanks are enclosed in an outer shell and buried in a fireproof material such as sand or pearlite.

FIGURE 1.8
EPSDU Site, Layout

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- The chlorosilane storage tanks are fireproofed, curbed, and remotely located at least 75 feet from the nearest flame.

- The process gantry, hydrogenation and distillation pump skids are a minimum of 25 feet from the nearest personnel operating position.

- Hydrogen storage is located remotely across the perimeter road.

- The hot-oil furnace and waste burners are remote from personnel and flammable fluids.

- Safety showers and eye washes are located on the ground level close to the process area and in the process gantry.

- The pyrolysis/melting/consolidation building, because of the presence of hydrogen, is ventilated a minimum of six air changes per hour and heated with flameless heaters (hot oil).

- The QC laboratory trailer has direct access to the pyrolysis/melting/consolidation building and is ventilated through hoods.

- Emergency power is supplied by a motor generator to assure maintenance of the refrigeration system.

- The oil filled transformer is isolated from adjacent buildings by two concrete block walls.

- Because of the presence of flammable gas, certain areas have been electrically classified as follows:

     Class 1, Division 2, Group B
          Hydrogenation pump skid
          Process gantry
          Silane shift tanks
          Pressure relief catch tank
          Liquid burner surge tank and agitator

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Class 1, Division 2, Group D

> Distillation pump skid
> Chlorosilane storage tanks
> Fuel oil storage tank (underground)
> STC recycle vaporizer

Four perspective sketches (Figures 1.9 through 1.12) of ten key EPSDU areas were prepared as visual aids for the Construction Design Engineering Group. The depicted areas are: M-G silicon hopper, gantry silane storage tanks, STC storage tanks, pumps, hydrogen compressors, silane pyrolysis building, Q.C. trailer, other trailers, and the waste treatment system.

1.1.2.2   Environmental and Safety

The environmental effort included the assessment for regulatory compliance of all environmental considerations associated with the process, preparation of standards, and obtaining appropriate approvals and permits. The safety aspects included evaluation of the process and detail design and monitoring of installation and operation to ensure that all features necessary for public and staff safety are included and proper procedures are used.

An Environmental Engineering Scope Design analysis was conducted which included site factors, applicable regulations and treatment of waste streams. The conclusions were as follows:

- Analysis of site sub-soil samples indicated the presence of oil and grease plus minor and non-hazardous concentrations of polychlorinated biphenyls (PCB's).

- Air-quality-wise the site is classified as a non-attainment area for particulates. The Control Agency (East Chicago Department of Air Quality Control) required mass balances on each pollution control device in order to evaluate permit requirements.

UCC
SILANE-TO-SILICON
EPSDU
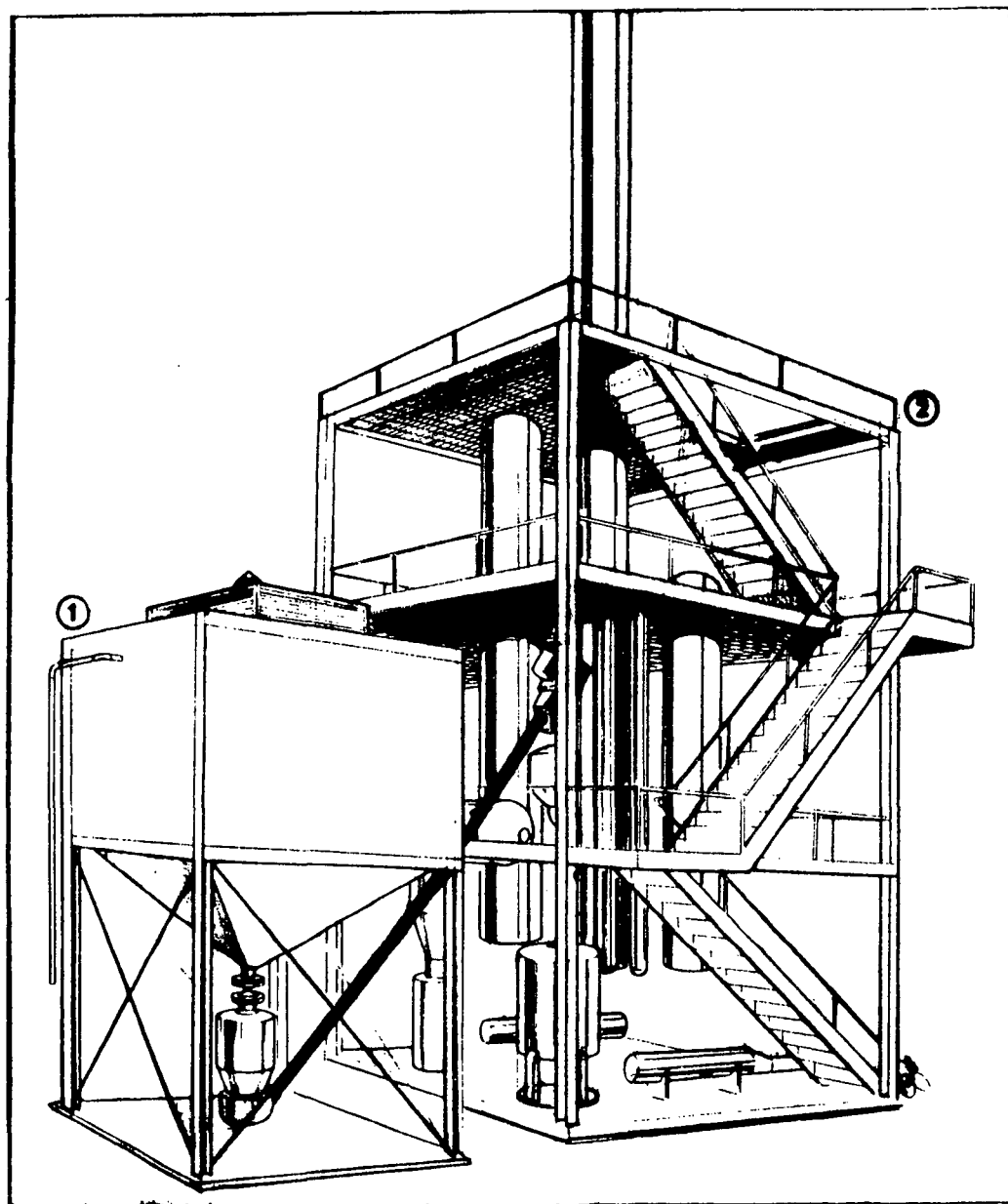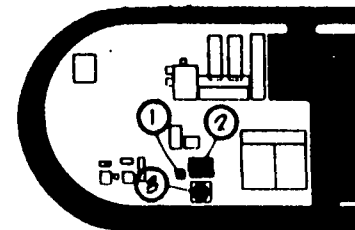
① CATALYST BLENDING TANK
FEED TRANSFER CONVEYOR
M.G. SILICON STORAGE BIN

② DISTILLATION COLUMNS
REDISTRIBUTION COLUMNS
CONDENSERS, REBOILERS
HYDROGENATION REACTOR

③ TANK ENCLOSURE
SILANE STORAGE TANKS
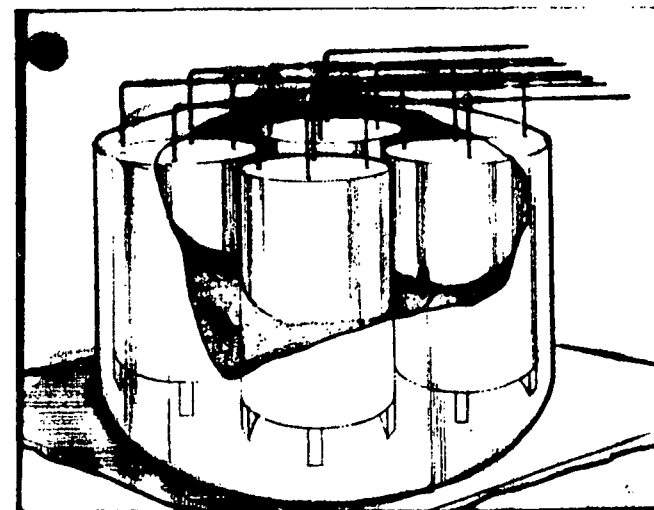
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FIGURE 1.9   Silane-to-Silicon EPSDU, Facility Sketch #1

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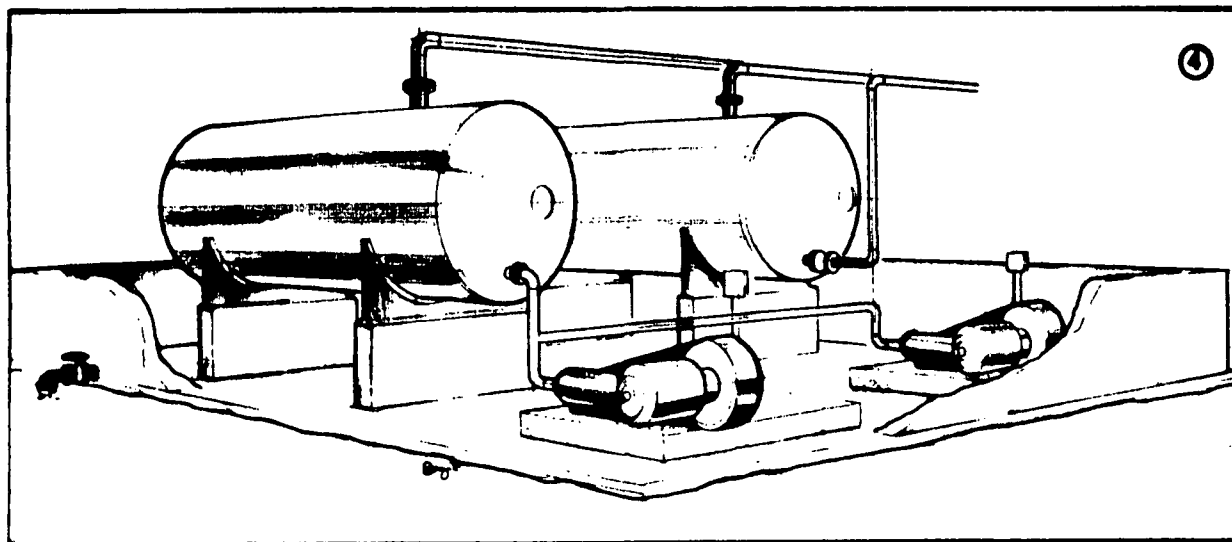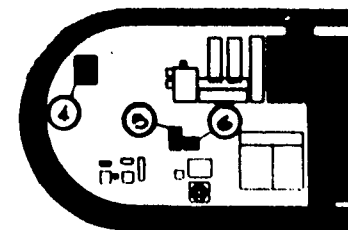
④ STC STORAGE TANKS
TRANSFER PUMPS
TCS STORAGE TANK

⑤ RECYCLE PUMPS
QUENCH PUMP
WASH PUMP
TRANSFER PUMPS

⑥ RECYCLE H2 COMPRESSORS
PYROLYSIS H2 COMPRESSOR
H2 RECEIVER COOLER

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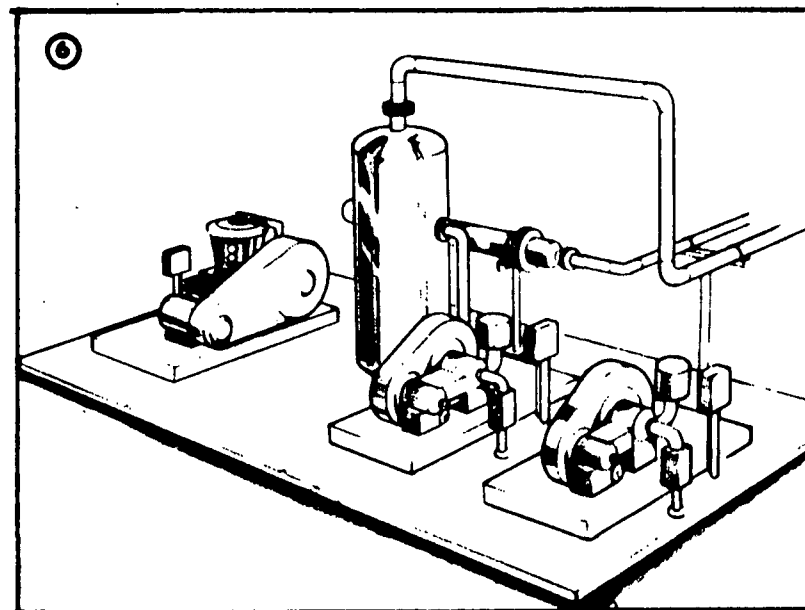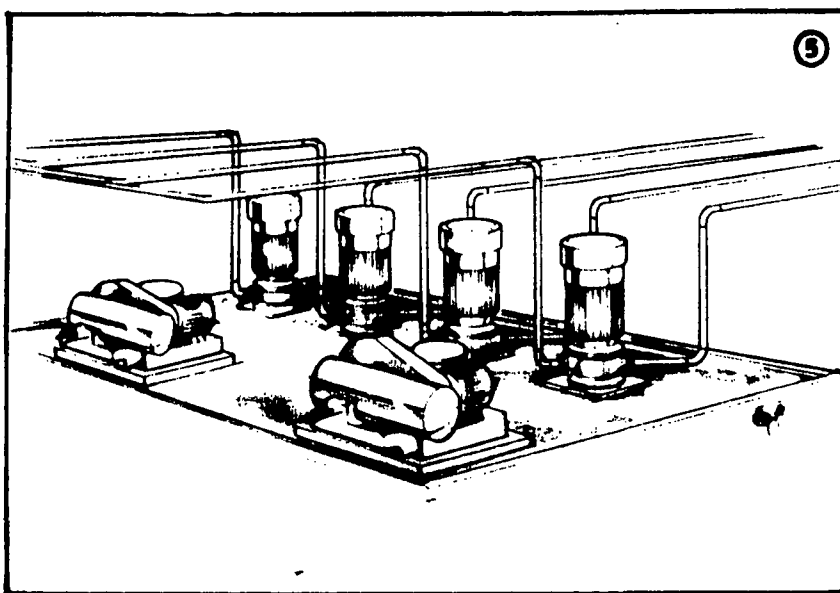
FIGURE 1.10   Silane-to-Silicon EPSDU, Facility Sketch #2

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EPSDU
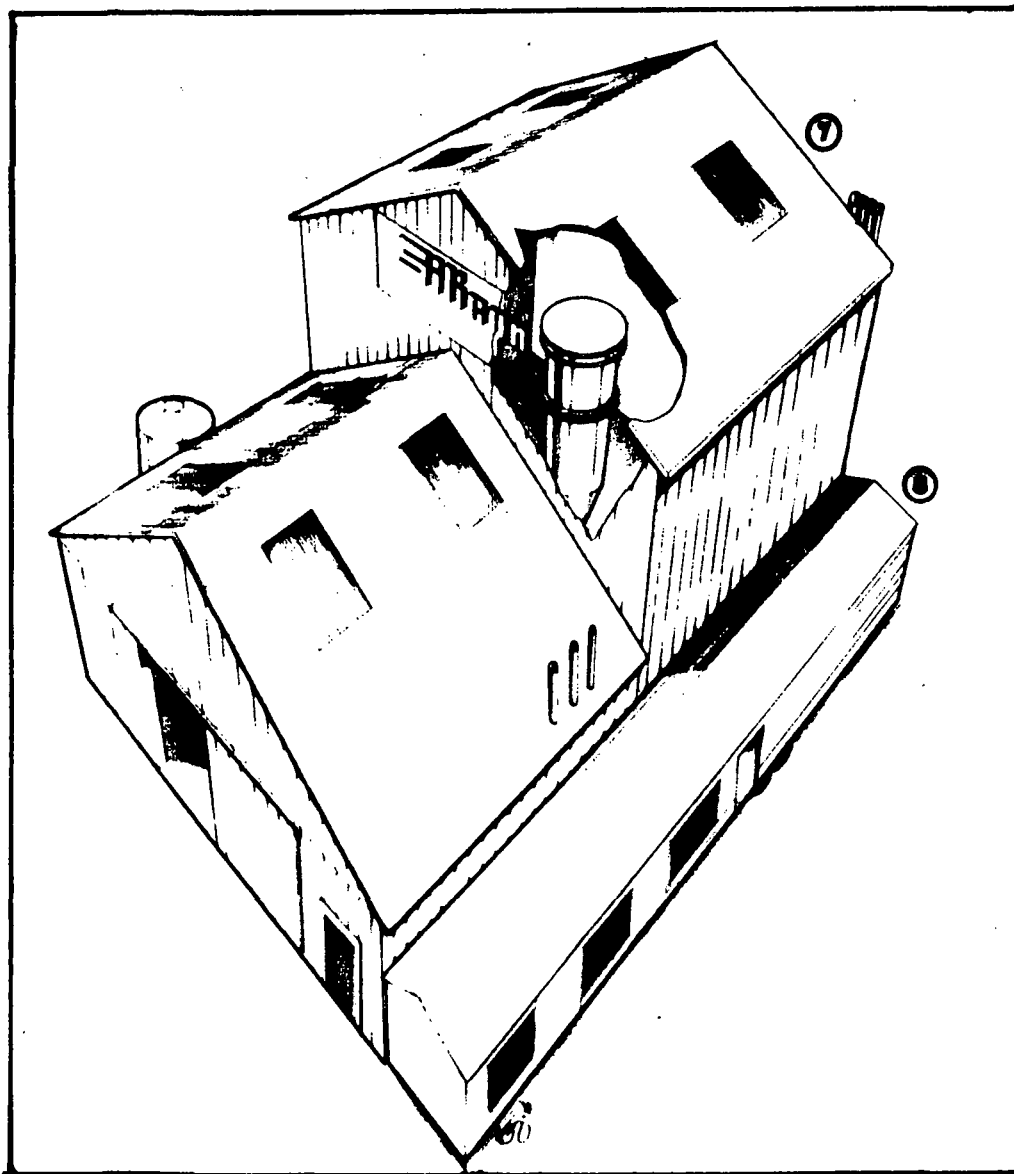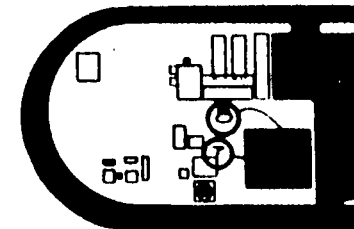
UNION CARBIDE

⑦ PYROLYSIS REACTOR
SILICON STORAGE BIN
SILICON SHOTTING TOWER

⑧ ANALYTICAL LABORATORY TRAILER

FIGURE 1.11 Silane-to-Silicon EPSDU, Facility
Sketch #3
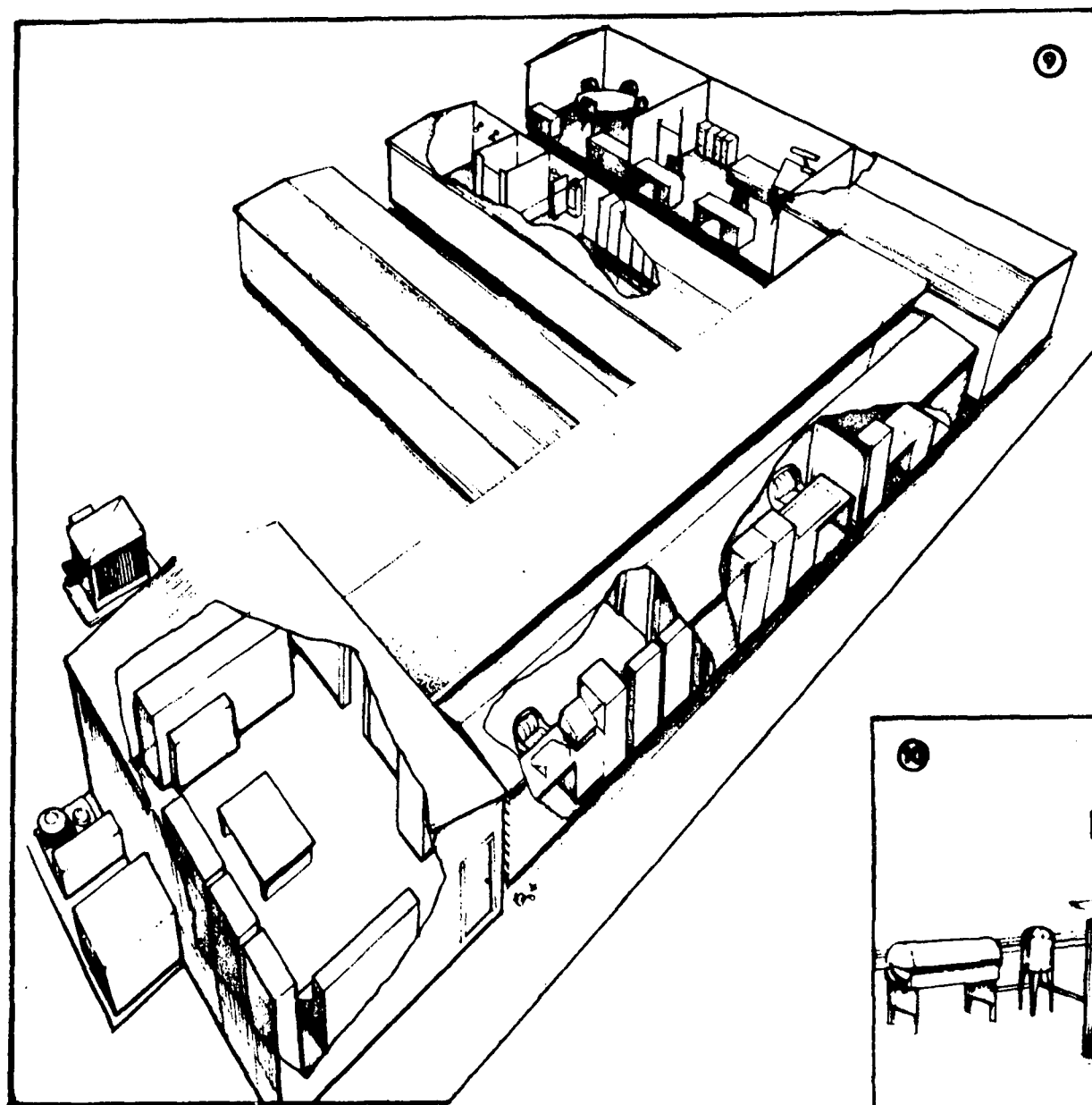
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EPSDU
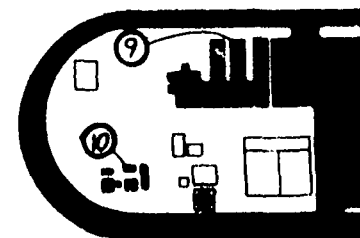
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⑨ ELECTRICAL / PROCESS CONTROL ROOM
ENGINEERING SUPPORT TRAILER
OPERATIONS SUPPORT TRAILER

⑩ WASTE GAS / LIQUID BURNERS
WASTE SOLIDS FILTER
WASTE GAS SCRUBBERS
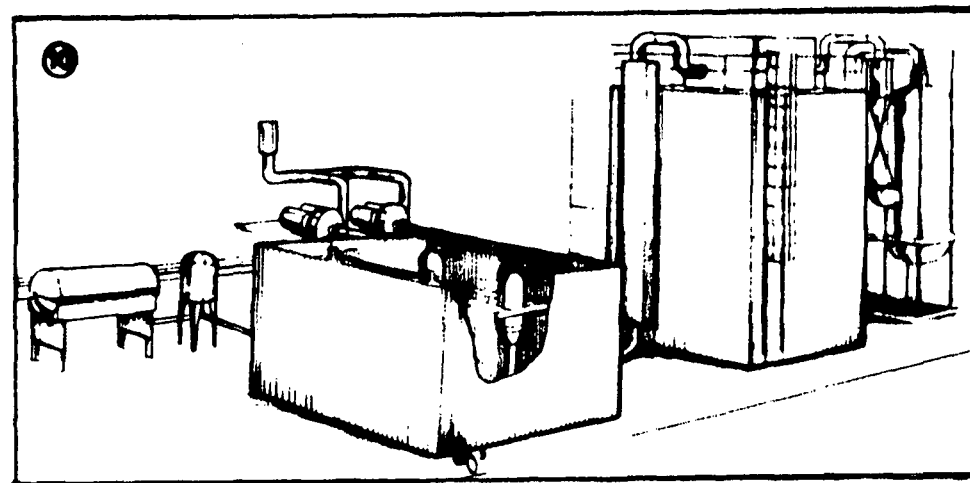
FIGURE 1.12   Silane-to-Silicon EPSDU, Facility Sketch #4

- The East Chicago Sanitary Sewer District required detailed information on trace metal contaminants and priority toxic pollutants that are expected to be in the waste water. A modification to the existing permit to discharge into the POTW was required from the regulatory agency.

- Treatment of wastewater to neutralize acid will utilize established Best Available Economically Achievable for similar industrial SIC categories.

- The facility will generate a small quantity of hazardous residues. The hydrolysis of chlorosilanes inadvertently released to the air will form irritating HCl vapors which could create environmental and occupational health problems.

- Incineration of waste chlorosilane gases and liquids, followed by removal of combustion gas solids and acid vapors, involves some degree of process performance risk.

- The physical and chemical properties of chlorosilanes and their combustion products required that these substances be engineered away from the work-place environment.

- The reactive nature of hydrochlorosilanes and silane can lead to the formation of hydrogen in closed sewer systems which can create a hazard if adequate precautions are not taken. Silane and chlorosilanes can ignite spontaneously when released to the atmosphere.

Based on the conclusions, the following recommendations were developed:

- Prior to construction, about ten core borings should be taken at depths of approximately 3, 6, and 12 feet to determine if there is any significant sub-surface soil contamination of other environmental problems.

- The air pollution control devices and treatments are summarized in Table III with estimated treatment efficiencies

and emission rates. The dilute HCl from the scrubber is to be neutralized with caustic soda.

- All process areas shall be provided with a process sewer which discharges into a neutralization tank and, subsequently into the Specialty Gases Plant process sewer.

- Since liquid chlorosilanes are classified as hazardous chemicals, storage tanks containing these substances require curbs and/or dikes, in accordance with Section 311, Part 112 of the Water Act. These confinements can be earth dikes or simple curbs with a capacity of 130% of the largest tank within the dike.

- Sludge and other residues from plant operations are likely to be classified as hazardous and therefore should be containerized, stored, transported, and disposed of in accordance with guidelines covered by the Resource Conservation and Recovery Act (RCRA). The rules for RCRA are currently being promulgated, and will be available to operating personnel for plant startup.

- The chlorosilanes reactor area and liquid chlorosilane pumps shall be sloped to drain pump seal leaks, spills, or other chlorosilane losses to an open wastewater trench connected to the process sewer system. Open trench systems and the subsequent avoid trapping hydrogen formation of explosive mixtures.

- For compliance with OSHA on HCl and chlorosilane fumes (HCl ceiling TLV of 5 ppm by volume), a flexible-duct exhauster for process areas is recommended. Pump maintenance, chlorosilane spills, and sampling operations are potential sources of HCl fumes.

- Underground process wastewater sewers can be used to collect discharges from the diked caustic soda and chlorosilane storage tanks, laboratory, and waste treatment areas.

- A pH control system with automated caustic feed to the neutralizer is recommended. Appropriate alarms should be provided. An effluent flow measuring device with integrator is also required, as well as an automatic flow-proportional sampler.

- A Spill Prevention, Control, and Countermeasure Plan is required for this facility. This will require a limited investment for diking and contingency spill containment supplies. Dikes are required for the caustic soda and chlorosilane storage tanks, and a curb for the therminol heater system.

The various waste streams are identified in Figure 1.13, Emissions Survey Diagram (Dwg. SKC-127107B), and the recommended treatments are summarized in Table III. Items that merit special comment are as follows:

- Emission points A1, A3, and A4 are bag-house vents, and represent the best available treatment economically achievable to remove particulates. These filters are commercially available. Some maintenance and bag repair/replacement expenses should be anticipated.

- Emission point A5 represents ambient hot air with no treatment required. This emission could be utilized as a source of building heat during the cold months.

- Emission point A8 is the therminol heater stack. No treatment is required but the stack should be high enough to avoid occupational health hazards to plant operating personnel.

- The waste chlorosilane (both liquid and gas) burners merit special comment. Although silicon tetrachloride and trichlorosilane mixtures are burned commercially to produce fume silica as a product, the conditions of combustion require careful control to achieve complete combustion and
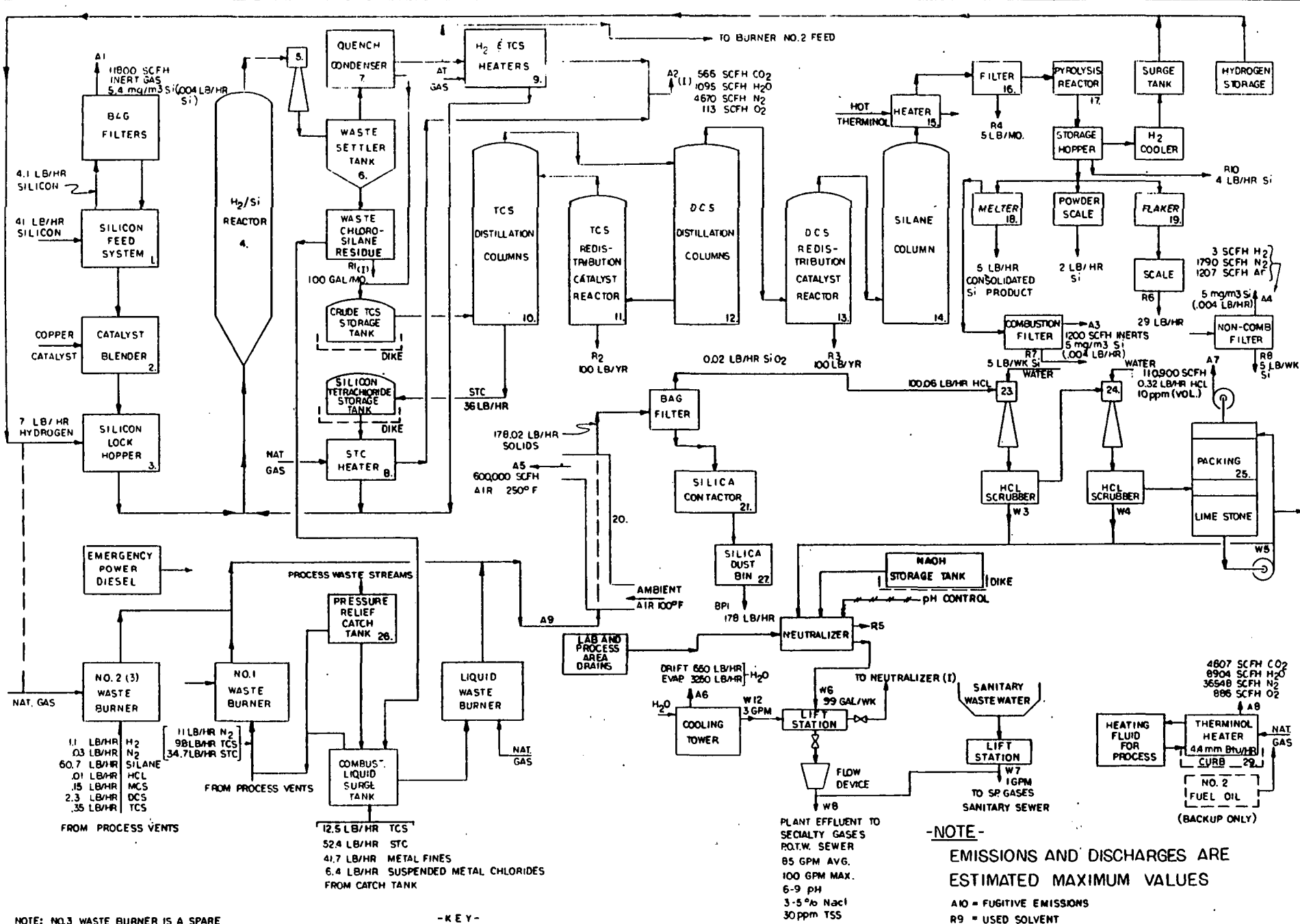
FIGURE 1.13 Emissions Survey Diagram

-KEY-

A = AIR EMISSIONS
BP = BY PRODUCT
W = WATER DISCHARGES
R = RESIDUE
N = ENVIRONMENTAL NOISE

MCS = MONOCHLOROSILANE
DCS = DICHLOROSILANE
TCS = TRICHLOROSILANE
STC = SILICON TETRACHLORIDE
(I) = INTERMITTENT

NOTE: NO.3 WASTE BURNER IS A SPARE

-NOTE-

EMISSIONS AND DISCHARGES ARE
ESTIMATED MAXIMUM VALUES

A10 = FUGITIVE EMISSIONS
R9 = USED SOLVENT

UNION CARBIDE CORPORATION
LINDE DIVISION
ENGINEERING DEPARTMENT • TONAWANDA, NEW YORK

EMISSIONS SURVEY DIAGRAM
SILICON EPSDU - PROJECT 7001
(PRELIMINARY)

SKC-127107

TABLE III EPSDU POLLUTION CONTROL (SHEET 1 OF 5)

SOURCE NO.	DESCRIPTION	UNCONTROLLED EMISSIONS OR DISCHARGES	RECOMMENDED TREATMENT	CONTROLLED EMISSIONS OR DISCHARGE	% REMOVAL EFFICIENCY
A1 (I)	Granular Silicon storage and feed bin (106)*	Intermittent, 1-2 times/mo several hours each time. 4.1-lb/Hr silicon (1.9 Kg/Hr)	Bag house	11,800 SCFH air (334 M^3/Hr) 0.004 lb/Hr (0.002 Kg/Hr) } Si 5 mg/M^3 (Concentration)	99.9
A2 (I)	Heater combustion gas vent; 0.7 MM Btu/Hr input. (136)	Infrequent vent. Inerts from buring natural gas in STC and recycle H_2 & TCS heaters. 6444 SCFH inerts (182.5 M^3/Hr)	None (is re-used in process)	Not applicable (NA)	NA
A3	Combustible gas filter vent from pyrolsis purge (400) (2200)	1200 SCFH max. argon, with a trace of H_2, plus 3.9 lb/Hr silicon (1.8 Kg/Hr)	Bag house	1200 SCFH, (34 M^3/Hr) argon with trace of H_2, 0.004 lb/Hr Si, (0.002 Kg/Hr) 5 mg/M^3 Si (Concentration)	99.9
A4	Non-combustible gas vent from filter. (452) (2300)	3000 SCFH max. inerts plus a trace of H_2. (85 M^3/Hr) 0.4 lb/hr Si	Bag house	3000 SCFH inerts (85 M^3/Hr) 0.004 lb/Hr Si (0.002 Kg/Hr Si) 5 mg/M^3 Si (Concentration)	99.9
A5	Air vent from agglomerator (414)	600,000 SCFH ambient air (16,992 M^3/Hr) Temperature 250°F (121 °C)	None - no contamination	600,000 SCFH (16,992 M^3/Hr)	NA
	* Process stream catalog number (I) = Intermittent				

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TABLE III EPSDU POLLUTION CONTROL (SHEET 2 OF 5)

SOURCE NO.	DESCRIPTION	UNCONTROLLED EMISSIONS OR DISCHARGES	RECOMMENDED TREATMENT	CONTROLLED EMISSIONS OR DISCHARGE	% REMOVAL EFFICIENCY
A6	Cooling tower drift and evaporate	650 lb/Hr drift (295 Kg/Hr) 3250 lb/Hr evaporate (1474 Kg/Hr) (primarily water)	None	650 lb/Hr drift (295 Kg/Hr) 3250 lb/Hr evaporate (1474 Kg/Hr)	N/A
A7	HCl Venturi and tails scrubber	110,900 SCFH inerts (3140 M^3/Hr) 7000 ppm x vol. HCl	No further treatment required	110,900 SCFH inerts (3140 M^3/Hr) 10 ppm x vol. HCl	99.9
A8	Therminol heater stack. 4.4 MM Btu/Hr	50,945 SCFH combustion gas from burning natural gas. (1443 M^3/Hr)	None	50,945 SCFH inerts (1443 M^3/Hr)	
A9	Combustion gases from waste liquid and gas burners.	110,900 SCFH (3167 M^3/Hr)	Discharged to agglomerator bag filter, venturi scrubbers, and packed scrubbers (see A7)	110,900 SCFH (3167 M^3/Hr)	99.9
A10 (1)	Miscellaneous workplace fumes, mainly HCl (OSHA type consideration)	Intermittent 0-100 ppm x vol. (Concentration)	Use portable fume exhauster to remove vapors from workers, and discharge through elevated stack.	Workplace environment less than 5 ppm x vol. HCl (Concentration)	90
W2	Cooling tower blowdown Phosphonate/phoshate type treatment	3 gpm (11.4 1/min.)	None. Discharge to process wastewater lift station	3 gpm (11.4 1/min.)	NA

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TABLE III EPSDU POLLUTION CONTROL (SHEET 3 OF 5)

SOURCE NO.	DESCRIPTION	UNCONTROLLED EMISSIONS OR DISCHARGES	RECOMMENDED TREATMENT	CONTROLLED EMISSIONS OR DISCHARGE	% REMOVAL EFFICIENCY
W7	Sanitary wastewater for 15-20 people	sewage plus washwater 1 gpm ave. 3.8 l/min.	Discharges directly to specialty gases sanitary sewer by separate lift station	Sewage plus washwater 1 gpm ave. 3.8 l/min.	NA
W1	Laboratory and process area trench wastewater	10-50 gpm containing 0-1% HCl and chlorosilanes (38-190 l/min.)	To Neutralizer	10-50 gpm wastewater in pH range of 6-9. (38-190 l/min.)	NA
W6	Aqueous neutralizer effluent	99 gal/week neutralizer wastewater (378 l/min.) pH 6-9, 3-5% HCl	Discharge to lift station	99 gpm neutralized wastewater (378 l/min.) pH 6-9 3-5% NaCl	100% of acid removed
W3,4,5	HCl Scrubber Process Wastewater	50 gpm wastewater 190 l/min.)	To neutralizer	50 gpm 190 l/min.)	
W8	Final silicon EPSDU effluent from wastewater lift station	85 gpm daily average (323 l/min.) 100 gpm daily maximum (379 l/min.)	(already treated) Discharge through Specialty Gases sewer line to POTW. Measure flow and sample period-ically.	85 gpm daily average (323 l/min.) 100 gpm daily maximum (379 l.min.) treated wastewater	

TABLE III EPSDU POLLUTION CONTROL (SHEET 4 OF 5)

SOURCE NO.	DESCRIPTION	UNCONTROLLED EMISSIONS OR DISCHARGES	RECOMMENDED TREATMENT	CONTROLLED EMISSIONS OR DISCHARGE	% REMOVAL EFFICIENCY
R1 (1)	Waste chlorosilane residue tank	Infrequent (plug ups only) approximately 35 lb/Hr waste chlorosilanes plus small quantity of silicon and copper fines	Transfer to chlorosilane catch tank for incineration	Wastewater with pH 6-9 100 gal/mo. (380 l/mo.)	
R2 (1)	Disproportionation reactor	100 lbs/year catalyst (227 Kg/year)	None Containerize for approved disposal	NA	NA
R3 (1)	Disproportionation reactor	100 lbs/year catalyst (227 Kg/year)	None Containerize for approved disposal	NA	NA
R4 (1)	Silane ultrafilter	5 lb/mo. silicon fines (2.3 Kg/mo.)	None	NA	NA
R5 (1)	Neutralizer sludge	200 lbs/week wet gel solids (9.1 Kg/week)	Non-hazardous neutralized sludge contains less than 0-1% copper. Containerize for approved disposal	NA	NA
R6	Flaker	Silicon metal (29 lb/Hr) 13.2 Kg/Hr.	Containerize for storage and approved disposal	NA	NA
R7 (1)	Combustibles filter	5 lb/week silicon metal (2.3 Kg/week)	None	NA	NA
R8 (1)	Non-combustibles filter	5 lb/week silicon metal (2.3 Kg/week)	None	NA	NA
R9 (1)	Used solvent from catalyst treatment	25 gal.year of hexane, toluene solvent (95 l/year)	Containerize as hazardous waste for approved disposal under RCRA regulations	NA	NA
R10 (1)	Waste Silicon Metal	4 lb/Hr, 1.8 Kg/Hr.	none	NA	NA

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TABLE III EPSDU POLLUTION CONTROL (SHEET 5 OF 5)

SOURCE NO.	DESCRIPTION	UNCONTROLLED EMISSIONS OR DISCHARGES	RECOMMENDED TREATMENT	CONTROLLED EMISSIONS OR DISCHARGE	% REMOVAL EFFICIENCY
BPl [1]	Silica dust bin (SiO_2)	178 lb/Hr. silica (81 Kg/Hr.)	None	NA	NA
SPCC Require-ments	Dike for STC tank Dike for TCS tank Dike for NaOH tank	Chlorosilane spills NaOH spills	Contain and neutralize "	Neutralized wastewater with some solids	NA
	(1) BP = By Product (2) NA = Not Applicable STC = Silicon Tetrachloride TCS = Trichlorosilane				

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avoid the formation of free chlorine or phosgene ($COCl_2$), both of which are toxic chemicals. Because of the potential lack of sufficient hydrogen in the burner feed stream to convert all of the chlorine to HCl, additional hydrogen is usually required. The stoichiometry of the required reactants is presently being developed. In addition, the formation of fume silica (SiO_2) during the combustion process often results in a build-up of silica solids on the burner walls which requires frequent mechanical removal to sustain satisfactory burner operation.

- Particle size of the fumed silica formed in combustion is exceedingly small (0.1 to 1 micron) which is too fine for efficient removal from the gas stream by filtration or wet scrubbing methods. To increase removal efficiency, the fume silica is passed through an agglomerator, which is essentially an air-cooled heat exchanger that promotes particle growth of the fumed silica.

- Removal of HCl vapor in the combustion gases is accomplished by a two-stage, venturi scrubber system, followed by a packed tail-gas scrubber. The resulting HCl absorbed is neutralized with caustic soda. Suspended solids in the neutralized effluent are expected to range from 50 - 100 ppm total suspended solids. Emission point A8 from the HCl tail-gas scrubber can be expected to have a stack HCl concentration of 10 ppm by volume, which is acceptable to the local regulatory agency.

- Wastewater discharges are expected to range from 85 - 100 gpm. Of this quantity, approximately 20 gpm is derived from cooling water discharges. Annual reconditioning of the disproportionation catalyst will generate several hundred gallons of organic solvents, such as toluene and methanol. Occassional discharge of a small quantity of acqueous methanol to the process sewer can be expected. The used toluene will be containerized for approved disposal according to RCRA requirements.

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- The neutralizer will require occasional cleaning and re-moval of sludge and sand. Access flanges at the top are recommended to facilitate this operation.

- The gantry should be diked/curbed to contain oil leakage and potential spills. An earth or concrete dike could be considered. A pipeline and valve is needed to drain the dike.

- The sanitary wastewater system is to be provided with its own lift station and pump system. One source of this type of specialized equipment is the Gould Pump Company.

- The East Chicago Air Quality Control Office may require occasional stack/vent sampling to determine if we are in compliance with our permit. This may incur $5,000 - $10,000 expense after startup. A requirement for periodic waste-water analysis can also be expected, with special attention being directed to the concentration of copper and total suspended solids (TSS) in the effluent.

A water discharge permit was received from the East Chicago Sanitary Sewer District. The permit allowed us to initiate necessary con-struction work and also stated that we would be required to provide sampling equipment to collect representative samples of our discharge stream, and a flow metering device to record total process flow discharge into their system.

Fourteen design safety checklists, used as design standards by specific engineering disciplines, were reviewed for EPSDU design applica-bility. The following ten checklists are applicable:

- Electrical Power System   (SA-5-2)
- Instrumentation   (SA-5-3)
- Distillation Columns   (SA-5-5)
- Mechanical Equipment   (SA-5-6)
- Facility Layout   (SA-5-9)
- Architectural Construction Design   (SA-5-10)
- Mechanical Construction Design   (SA-5-11)

- Electrical Construction Design   (SA-5-12)
- Instrumentation Construction Design (SA-5-13)
- Auxiliary Equipment   (SA-5-16)

These checklists were utilized during final design reviews as each design activity was completed.

Safety Reviews

A design review team was established which has the responsibility of considering all aspects of construction and operation of the EPSDU facility from a safety viewpoint.

The team is multi-disciplined, organizationally disinterested, and technically highly qualified in the following disciplines: fire protection, personal safety, operations, engineering, and process design/chemistry. Although possessing these multifunctional skills, the team will consult other specialists to give the greatest possible depth to the safety review. Initially, the team concentrated on the design but will reconvene as a pre-startup review team at the 95% construction completion stage. The initial meeting of the team took place at Tonawanda on November 18 - 20, 1980.

The safety review team, reviewed the design basis for the facility, materials of construction, layout, control methodology, and emergency equipment and procedures. The emphasis was placed on process and personnel safety. The review included safety and operability of the M-G silicon-to-silane portion of the process and features of the EPSDU site layout, process equipment, and fire protection. The safety team comments were subsequently reviewed by engineering personnel and the suggested items were implemented in the EPSDU design.

The air discharge permit application was approved as a permit-to-construct by the Indiana Air Quality Office for East Chicago.

The engineering design of the EPSDU addresses all safety and environmental concerns about process wastes, neutralization of chlorosilane liquid spills, diked areas, drainage trenches, fire-fighting techniques and hydrant and protective equipment.

Volume I of the Operations Manual describes the handling of plant chemicals and personnel safety procedures.

1.1.2.3   Gantry Scale Model

A scale model of the gantry (3/4 inch equals 1 foot) was constructed to serve as a tool to assist the mechanical designers during drawing checkout; see Figure 1.14. The structure, major equipment and piping runs were identified and color-coded. The model will be used during the mechanical bid phase, operator training and maintenance planning. The use of the model had shown some interferences in piping runs which would have been costly to treat as a field modification.

1.1.2.4   Operations Liaison

This activity covered the coordination with the UCC East Chicago Gas Plant in order to locate the EPSDU site adjacent to the gas facility and provide "tie-ins" to handle wastewater flows and necessary process gas supplies (nitrogen, etc.). Operations personnel participated with engineering designers in locating roadway access, site preparation, fencing, construction and sub-contractor access and parking. They also provided plant-site coordination during the bidding phase and assisted EPSDU installation activities by providing on-site day-to-day construction supervision of the site preparation work.

1.1.2.5   Facility Scale Model

The facility scale model was started but not completed during Phase III. The model preparation was discontinued due to the proposal that the EPSDU site be relocated.

1.1.3   EQUIPMENT DESIGN, SPECIFICATION, PROCUREMENT

The equipment-related effort included development of the control system, preparation of the piping and instrumentation diagram, preparation of wiring schematics and control panel drawings, and the design of equipment. The specification activity included definition of specific requirements for
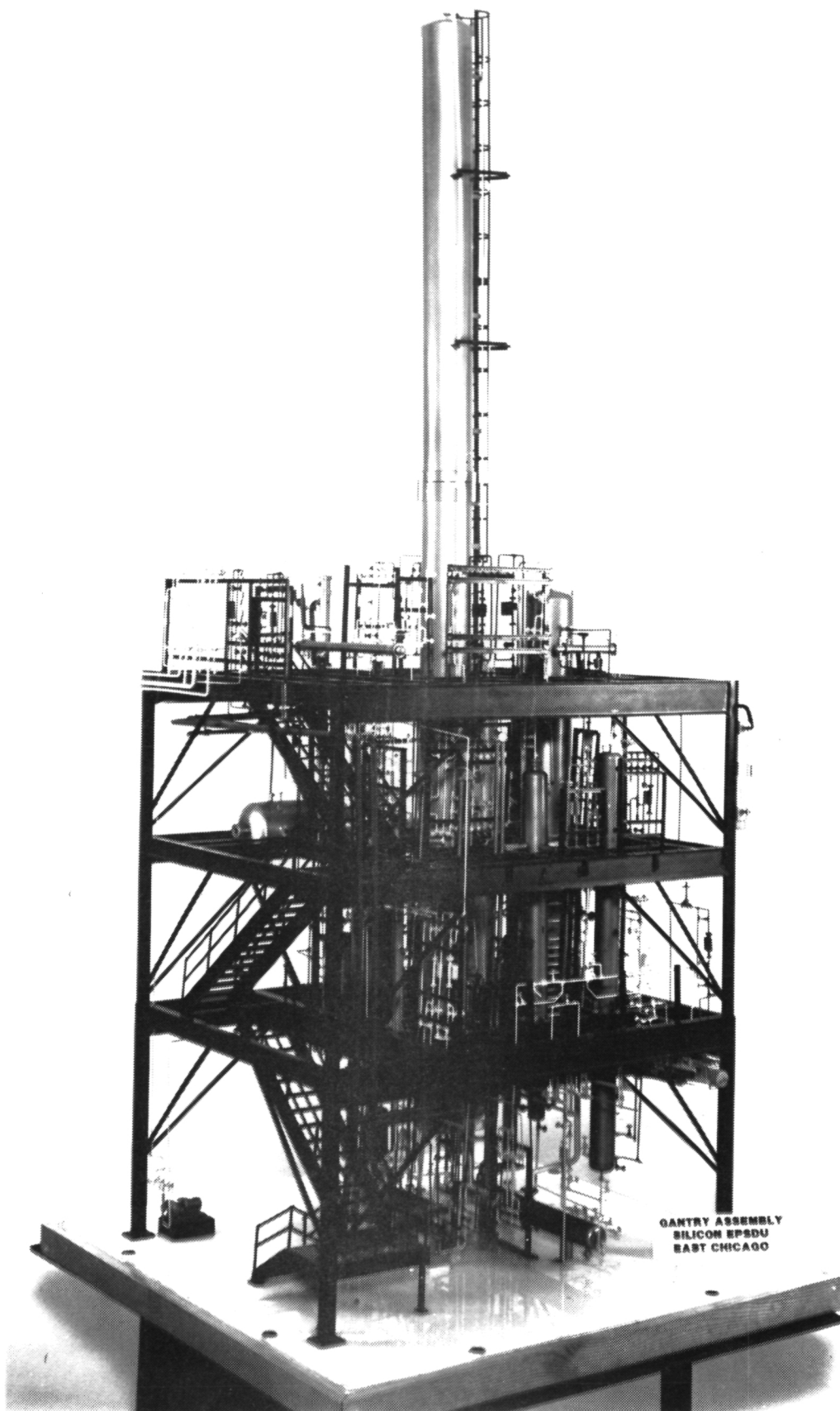
FIGURE 1.14 Gantry Scale Model

each item of equipment, preparation of bid packages, evaluation of vendor quotation, and preparation of final specifications and drawings. Procurement included the issuance of procurement packages to selected vendors and obtaining comprehensive design information necessary for preparing installation drawings.

The design and procurement of each item of equipment was accomplished through the combined efforts of process engineers, equipment engineers, and purchasing agents. These efforts produced a series of documents that evolve, ultimately, into a complete, definitive procurement package. The guidelines for the individual documents and their respective uses are as follows:

- Functional Specifications: This specification is developed by the process engineer based on process requirements reflected in the process flow diagram, heat/mass balance, and process control scheme. It defines the duty that this item of equipment must perform for the overall process system to operate.

- Engineering Specifications: Using the Functional Specification as a basis, the equipment engineer determines the specific type of equipment necessary to satisfy the process requirements. This translation of process requirements into hardware-specific information is delineated in the engineering specification.

- Request for Quotation (RFQ): The Request for Quotation, prepared by the equipment engineer, summarizes the equipment requirements, identifies vendors to be contacted, and defines the bidding instructions. This form, plus the engineering specification, constitute the RFQ package submitted to the purchasing department for transmittal to potential vendors. Vendor quotations are reviewed by the equipment engineer and, based on a technical, cost and scheduled evaluation, a vendor is selected.

- Request for Requisition (RFR): The Request for Requisition, prepared by the equipment engineer, consists of the RFQ

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package plus the Bid Evaluation Report that identifies the selected vendor and the specific equipment model to be purchased. The RFR is submitted to purchasing and serves as the technical basis for the purchase order.

- Purchase Order (PO): The Purchase Order (Form L334-31D0), prepared by the purchasing agent, defines the terms and conditions, delivery requirements, and billing instructions applicable to the particular equipment and vendor. This form supplements the technical information contained in the RFR to provide a complete procurement package. When the internal review cycle is completed and the appropriate approvals have been obtained, the validated Purchase Order is issued to the vendor.

- Procurement Status Report (PSR): After the order is placed, technical performance is monitored by engineering personnel and contractual performance is monitored by purchasing personnel. Status is reflected in the Procurement Status Report, which is included in the Appendices.

These six documents serve as milestones for measuring performance of the procurement cycle for each item of equipment.

A list of major equipment items (100) is included in the Appendices (see Appendix A).

1.1.3.1   Process Control

The control systems engineering effort included all activities associated with developing P&I diagram, designing process control loops and control panels, specifying valves and instrumentation, and preparing control wiring and pneumatic tubing diagrams.

The "product" of the process control system design activity was a series of formal documents or drawing packages which define individual

instrumentation requirements, process design requirements, equipment require-
ments, and piping requirements in great detail. These documents were used
by all other engineering groups for their design activities and will also be
used by construction and operations personnel in the future. The key docu-
ments are:

D-2143505 - Process & Instrumentation Diagram (50 sheets)

D-2146786 - Schematic Wiring Diagram (17 sheets)

D-2145969 - Logic Diagram (18 sheets)

A-2145987 - Field Instrumentation Equipment List -
Functional Specs

A-2146764 - Field Instrumentation Equipment List -
Detailed Specs

A-2145988 - Main Instrument Panel Specifications

A-2145989 - Quality Assurance Panel Specifications

A-2147839 - Local I/O Panel Specifications No. 1

A-2147840 - Local I/O Panel Specifications No. 2

A-2147841 - Local I/O Panel Specifications No. 3

A-2147842 - Local I/O Panel Specifications No. 4

A-2145970 - Automatic Valve Specifications

The selection of individual instrumentation components such as
automatic valves, process controllers, and piping components was difficult.
Each item had to be specified and evaluated for functioning within the harsh
chlorosilane environment. The necessary in-depth operability and safety
evaluations, consultations with equipment suppliers, and in-house reviews
was time-consuming.

The following instrumentation components will be mounted on the
main control panel:

● Alarm-Shutdown monitors - 4 items

- Alarm-Shutdown system components - 51 items
- Electrical switches - 27 items
- Pressure gauges - 5 items
- Pressure controllers - 10 items
- Flow controllers - 11 items
- Level controllers - 7 items
- Temperature controllers - 6 items
- Ratio controller - 1 item
- Manual controllers - 3 items
- Strip-Chart recorders - 4 items
- Temperature transmitters - 6 items
- Orifice plates and flow sections - 5 items
- Flowmeters and transmitters - 48 items
- Differential pressure transmitter - 1 item
- Pressure switches - 7 items
- Flowmeter - 1 item
- Flow indicators - 8 items
- Valve manifolds - 18 items
- Flow and level indicators - 12 items
- Pressure and temperature switches - 23 items
- Differential pressure transmitters - 20 items
- Conductivity-type level indicators - 15 items

A master instrument equipment list was developed from the P&I diagrams and the schematic wiring diagram was expanded to show the interfaces between field instruments, panel instruments and the data collection system.

P&I Diagram

The Process and Instrumentation diagram (P&I), which depicts all portions of the process except pyrolysis and melting/consolidation was issued. An instrumentation evaluation determined that most process fluid flows will

be measured by rotameter-type devices and most process liquid levels will be measured by capacitance probes. The detailed piping and valving arrangements required for these devices were depicted on the P&I diagram. Similarly, certain pieces of process equipment have been purchased with instrumentation supplied by the vendor. Items reviewed included safety relief valve settings, manual and check valve relative locations, pressure and temperature measurement locations, and automatic valve sizes.

The Revision "G" version of the P&I diagram was the last issue. It was used to check all equipment and piping drawings in the mechanical installation bid package. This was also the version of the P&I diagram to be issued prior to the actual start of equipment and piping installation work at EPSDU.

Control Logic Diagram

The control logic diagram and schematic wiring diagrams were prepared. Purchasing specifications, associated with the control logic diagram, were issued for the main programmable control system or "Modicon" unit. The main components of this system were shipped to Tonawanda for checkout and control logic programming.

Relay logic diagrams were developed to enable specification of the relay circuit equipment, and development of the schematic wiring diagram.

Modicon Programming

The Modicon 584 programmable controller is a solid-state device designed to perform logic, timing, sequencing and calculations for industrial control applications. It is a general purpose device that can be used as a direct replacement for relays or solid-state electronics in an industrial environment. This system is used for the EPSDU facility.

Control Panels

Figure 1.15 represents the design of the operating/front face of the main control panel that will be located in the switchgear-control room.

The EPSDU contains a total of six control panels.

1.1.3.2 Electric Power System

This effort included all electrical engineering activity required to develop the power control and distribution systems, prepare the definitive electrical one-line diagram (Figures 1.16 and 1.17) and specify electrical equipment. Alternates for a secondary source of emergency power were also evaluated and the most reliable source appeared to be a diesel-powered emergency generator at the EPSDU site. If primary power is lost, the emergency generator will automatically cut in and provide a level of power sufficient to operate the refrigeration system, some ventilation systems, and some lighting systems. Consequently, the emergency generator was purchased.

The 11.5 KV primary power will be supplied by NIPSCO and is reflected in the issued electrical one-line diagram. This diagram delineates, through single lines and simplified symbols, component devices and circuits that comprise the electrical system. It depicts the path of energy transfer and identifies circuit components and their ratings. Thus, the one-line diagram is a complete representation of the EPSDU electrical system from the receiving point to the equipment. Typical information includes:

- Power Supply - Incoming voltage, frequency and short cirucit . EPSDU incoming voltage is 11.5 KV, maximum short circuit is 36.4 MVA, and minimum short circuit is 24.7 MVA.

- Transformers - EPSDU includes a 1500 KVA, 11.5 KV/480 V power transformer with an impedence of 8.0%. Four 480 V to 120/ 280 V transformers are distributed throughout the plant for lighting, HVAC, and other general uses.

- Power Distribution Cable and Bus Ducts - Size and capacity

LIFTING LUG

FIELD WIRING ENTRY (EXCEPT POWER).

B

POWER WIRING ENTRY FOR CIRCUIT BREAKERS.

TERMINAL BLOCKS

MODICON MAIN FRAME

MODICON I/O

MODICON J200 I/O EXPANDER

LI 107-1 · FI 118 · FI 317-2 · FI 238-1 · FI 240-2 · FI 119-1 · FI 204-1 · FI 216-1 · FI 214-1 · FI 222-1 · FI 227-1 · TI 231-1 · TI 231-2 · TI 231-3 · TI 231-4 · PI 235-1 · FI 412-1 · FI 411-1 · FI 413-1 · FI 413-2

UI-3   UI-4   UI-1

TIC 1 · TIC 2 · LI 109-1

TI 207-1 · TI 217-1 · TI 451-1

AC 104-1 | LIC 116-1 | LIC 119-1 | HIC 118-1 | SPARE | PIC 312-1 | PIC 317-1 | PIC 317-2 | FIC 121-1 | SPARE

SPARE | SPARE | PIC 128-1 | FIC 128-1 | LIC 2000-1 | SPARE | SPARE | AR 118-1 | TR 107-1

FIC 125-1 | FFC 200-1 | PIC 250-1 | LIC 206-1 | PIC 213-1 | FIC 2002-1 | FIC 213-1 | TDIC 205-1 | LIC 238-1 | TIC 216-1

SPARE | PIC 214-1 | HIC 224-1 | PIC 230-1 | FIC 232-1 | TDIC 232-1 | FIC 200-1 | LIC 225-1 | HIC 240-1

AR 213-1 | AR 220-1 | HIC 214-1 | PIC 2003-1 | FIC 220-1 | TDIC 220-1 | LIC 212-1 | TIC 223-1 | SPARE

AR 212-1 | FR 227-1 | AR 200-1 | SPARE | LR 231-1 THRU LR 231-4 | FIC 233-1 | SPARE | SPARE | SPARE

PB7

PL1 · PL2 · PB1 · PL3 · PL4 · PB2 · PL5 · PL6 · PB3 · PL7 · PL8 · PB4 · PB5 · PB6 · PL9 · PL10 · PL11

CIRCUIT BREAKER BOX "CP1"

7½'

14'

FRONT VIEW

B

UNISTRUT

SUBPANEL

6" CHANNEL IRON BASE (4 SIDES)

2½'

SECTION BB

FIGURE 1.15

PANEL LAYOUT

UNION CARBIDE CORPORATION
LINDE DIVISION
ENGINEERING DEPARTMENT · TONAWANDA, NEW YORK

MAIN INSTRUMENT PANEL "A"

480 VOLT MOTOR CONTROL CENTER #1 – SIDE A – NEMA 1

480 VOLT - 3 ∅ - 60 HZ - 42,000 AIC - 1600 A. COPPER - SPLIT MAIN BUS

No.	Breaker	Description	Size
101	HFB-50	SPARE	
102	CS-1	GAS SUPERHEATER AIR BLOWER (429-04)	3
103	HFB-50	SPARE	
104	HFB-50	480 V FEEDER TO INSTRUMENT AIR PACKAGE (469-14)	
105	CS-1	SPARE	
106	CS-2	THERMINOL HOT OIL PUMP (466-02)	15
107	CS-1	THERMINOL AIR BLOWER (469-16)	3
108	CS-4	COOLING TOWER WATER PUMP #1 (466-04)	50
109	CS-3	COOLING TOWER FAN (469-02)	25
110	CS-2	STC PUMP (SPARE) (426-07)	10
111	CS-2	STC PUMP (MAIN) (426-06)	10
112	HFB-150	480 V. FEEDER TO PYROLYSIS REACTOR HEATER PACKAGE (445-02)	
113	HFB-150	SPARE	
114	HFB-150	PRODUCT MELTER #3 (449-08)	100 KW
115	HFB-150	PRODUCT MELTER #2 (449-06)	100 KW
116	HFB-150	PRODUCT MELTER #1 (449-04)	100 KW
117	CS-2	SPARE	
118	CS-3	PYROLYSIS HYDROGEN COMPRESSOR (443-02)	40
119	CS-2	RECYCLE HYDROGEN COMPRESSOR (SPARE) (423-03)	10
120	CS-2	RECYCLE HYDROGEN COMPRESSOR (MAIN) (423-02)	10
121	CS-4	SPARE	
122	CS-3	DCS DISTILLATE PUMP (436-04)	25
123	CS-4	TCS DISTILLATE PUMP (436-02)	75
124	CS-4	TCS & DCS QUENCH CONTACTOR PUMP (SPARE) (436-05)	75
125	CS-2	QUENCH CONTACTOR PUMP (MAIN) (426-02)	15

1600 A. SPLIT COPPER MAIN BUS

FOR OTHER SIDE OF 1600 A. SPLIT COPPER MAIN BUS, REFER TO MCC#1, SIDE B, DRAWING D-2145548 SHEET #2

Main feeder (item 100)

SPCB 2500
480 V. 2 PT'S 120 V.
2500 AMP - 600V - 3∅ 3 W - WITH ½ CAPACITY GND. - O/D - BUS DUCT
MAIN BREAKER
480 V.
1500 KVA - 3∅ 8.0% Z 55/65 °C
11.5 KV.
600 A FUSED LOAD BREAK SWITCH W/CLE-100 E FUSES
UNDERGROUND FEEDER SUPPLIED, INSTALLED, AND PROTECTED BY NIPSCO.

NIPSCO 11.5 KV FEEDER
MAX. S.C. = 36.4 MVA
MIN. S.C. = 24.7 MVA
NIPSCO WILL CHANGE TO 12.5 KV IN FUTURE BUT S.C. REMAINS THE SAME.

480 VOLT MOTOR CONTROL CENTER #1 — FLOOR PLAN

F	E	D	C	B	A
G	H	I	J	K	

480 VOLT MOTOR CONTROL CENTER #1 — SIDE A - FRONT ELEVATION

F	E	D	C	B	A
FUTURE	FUTURE	CS-1 102	CS-2 111	VS ⊕	
HFB-150 112	HFB-50 101	CS-1 107	CS-2 110	CS-2 125	SPCB 2500 100
HFB-150 113	HFB-50 103 104	CS-4 105	CS-2 106	CS-2 120	
HFB-150 114	CS-3 109	CS-3 118	CS-3 122	CS-2 119	
HFB-150 115	CS-4 108	CS-4 121	CS-4 123	CS-2 117	
HFB-150 116				CS-4 124	

10'-4"   90"

480 VOLT MOTOR CONTROL CENTER #1 — SIDE B - FRONT ELEVATION

K	J	I	H	G
∅ HMC 600 126	FUTURE CS-1 127	FUTURE	HFB-150 135	HFB-50 140 142
	CS-1 128	HFB-100 137	HFB-150 136	HFB-50 143 144
CS-3 131	CS-4 132	30 KVA XFMR. 138	HFB-150 141	HFB-50 145 146
CS-4 133	CS-4 134		HFB-150 151	HFB-50 147 148
			CS-1 129	HFB-50 149 150
			CS-1 130	HFB-70 139

8'-4"

∅ - UNUSABLE SPACE

FIGURE 1.16 EPSDU Electrical One-Line Diagram #1

480 VOLT - 3∅ - 60 HZ - 42,000 AIC - 1600 A. COPPER - SPLIT - MAIN BUS

(126) (127) (128) (129) (130) (131) (132) (133) (134) (135) (136) (137) (138) (139) (140) (141) (142) (143) (144) (145) (146) (147) (148) (149) (150) (151)

HMC600 | CS-1 | CS-1 | CS-1 | CS-1 | CS-3 | CS-4 | CS-4 | CS-4 | HFB-150 | HFB-150 | HFB-100 | HFB-70 | HFB-50 | HFB-50 | HFB-150 | HFB-50 | HFB-50 | HFB-50 | HFB-50 | HFB-50 | HFB-50 | HFB-50 | HFB-50 | HFB-50 | HFB-150

2 # 250 MCM THW/PHASE

WASTE GAS BURNER #1 (459-08) (1)
WASTE GAS BURNER #2 (459-10) (2)
WASTE GAS BURNER #3 (459-12) (2)
WASTE LIQUIDS BURNER (459-14) (1)
AGGLOMERATOR BLOWER (453-02) (30)
WASTE GAS INDUCTION BLOWER MAIN (453-04) (75)
WASTE GAS INDUCTION BLOWER SPARE (453-05) (75)
SPARE
SPARE

460 V. 75 KVA TRANSFORMER - 3∅ FLOOR MOUNTED IN CONTROL ROOM
120/208 V.

TRAILER #1 (OFFICE)   50A-3P 50A-3P TRAILER #2 (ENGINEERING)
TRAILER #3 (OPERATIONS) 50A-3P 50A-3P TRAILER #4 (MAINTENANCE)
FUTURE 50A-3P 50A-3P CONTROL BLDG. (HVAC ONLY)

120/208 V.-3∅-4W. 6 CKT. DIST. PANEL, LOCATED IN CONTROL BLDG.

480 VOLT FEEDER TO QUALITY CONTROL
LAB DISTRIBUTION PANEL (LOCATED IN THE MELTER BUILDING)
NORMAL POWER FOR QUALITY CONTROL LAB

TO 30 CKT. PANEL FOR QUALITY CONTROL LAB (LOCATED IN THE MELTER BUILDING)
120/208 V. 480 V.

480 VOLT FEEDER FOR YARD LIGHTING
QUENCH CONDENSER WASH PUMP (426-04) (1)
TO 480 V. DISTRIBUTION PANEL IN MELTER BUILDING (WELDING OUTLETS, CHAIN LIFTS, ETC.)
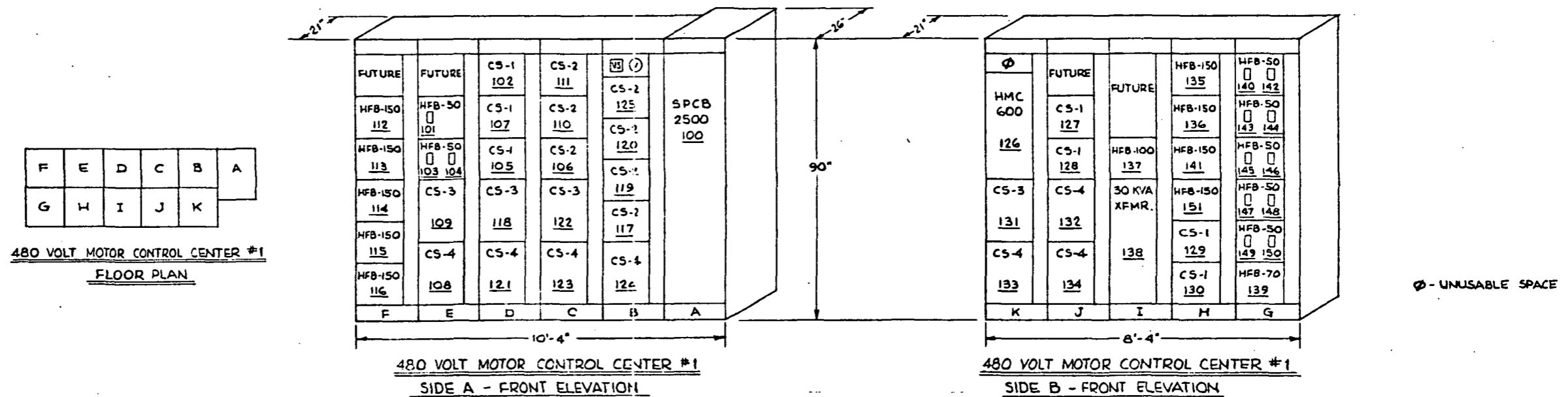WASTE SILICA DRUM PACKER (456-04) (1)
WASTE SUMP PUMP (466-08) (1)
SEWAGE LIFT PUMP (466-10) (1)
TAILING COLUMN PUMP (456-10) (1)
VENT FILTER SHAKER (457-04) (1)
WASTE LIQUIDS AGITATOR (459-16) (1)
TO 480 VOLT HEAT TRACING
SPARE
SPARE
SPARE

1600 A. SPLIT COPPER MAIN BUS

FOR OTHER SIDE OF 1600 A. SPLIT COPPER MAIN BUS REFER TO MCC #1 SIDE A, DWG. No. D-2145548, SHEET #1

480 VOLT MOTOR CONTROL CENTER #1 - SIDE B - NEMA 1

(200) (201) (202) (203) (204) (205) (206) (207) (208) (209) (210) (211)

480 VOLT - 3∅ - 60 HZ - 42,000 AIC - 600 A COPPER BUS

NORMAL FEED
400A-3∅-600 V. AUTOMATIC TRANSFER SW.
480 V. 2 PT'S 120 V.
VS (V)

CS-4 | CS-1 | CS-1 | CS-1 | CS-4 | HFB-50 | HFB-100 | HFB-100 | HFB-100

ALTERNATE FEED
EMERGENCY GENERATOR (300 KW)

REFRIGERATION COMPRESSOR (469-12) (75)
BRINE PUMP #1 (469-12) (5)
BRINE PUMP #2 (469-12) (5)
SPARE
COOLING TOWER WATER PUMP #2 (466-05) (50)
MELTER BUILDING VENTILLATION (5)

EM. LIGHTING & POWER
30 KVA-1∅ 120/208 V. 480 V.

EM. LIGHTING & CONTROL FOR Q.C.LAB.
30 KVA-1∅ 120/208 V. 480 V.
TO 30 CKT. PANEL FOR QUALITY CONTROL LAB (LOCATED IN THE MELTER BUILDING)

30 CKT. PANEL
1. CONTROL ROOM LIGHTS
2. REACTOR BLDG. LIGHTS
3. COMPUTER
4. MAIN INSTR. PANEL

480 VOLT MOTOR CONTROL CENTER #2 - NEMA 1

480 VOLT MOTOR CONTROL CENTER #2 FRONT ELEVATION

VS (V)	CS-1 202	HFB-50 206	∅
NORMAL 200	CS-1 203	HFB-100 207	30 CKT PANEL 209
	CS-1 204	HFB-100 210	
ALTERNATE	CS-4 201	30 KVA 1∅ XFMR.	30 KVA 1∅ XFMR.
	CS-4 205	211	208

∅ - UNUSABLE SPACE
* - THESE DIMENSIONS WILL VARY WITH THE MANUFACTURERS REQUIREMENTS.

90"   20" 32" 10" 20" 20" 20"   10'-2"

FIGURE 1.17 EPSDU Electrical One-Line Diagram #2

- <u>Bracing</u> - All equipment must be braced for short circuit.

- <u>Switch gear</u> - With protective relaying and equipment to control and protect large motors and transformers.

- <u>Motor Control Center</u> - With protective devices to control and protect smaller size equipment. EPSDU utilizes two motor control centers (MCC); however, MCC #2 is used to distribute power only to those items of equipment that must be powered during a power failure. A 300 kw generator is used for emergency power.

- <u>Motors & Heaters</u> - Horsepower and kw. EPSDU includes 33 motors ranging in size from 1 HP to 75 HP and three 100 kw heaters.

The one-line diagram serves as the basis for specifying the electrical equipment. Figures 1.16 and 1.17 (drawing D-2145548) show the electrical one-line diagram for the EPSDU project.

1.1.3.3   Process Equipment — UCC Designed

This included the in-house effort necessary to develop and prepare the complete design for specialized process equipment such as the hydrogenation and free-space reactors and distillation columns. UCC/Linde made the detailed mechanical design and manufacture of the four columns. The assembly drawings for the ASME-coded vessels were:

PS-2130700	-	Stripper Column Assembly
PS-2130702	-	TCS Column Assembly
PS-2130704	-	DCS Column Assembly
D&BM-2130706	-	Silane Column Assembly

A packed-bed silane column was selected rather than the originally conceived trayed column for ease in fabricating a very small distillation column.

A test was run on the silane distillation column packing to insure that the UCC cleaning procedure would not alter the packing or result in any unusual gas evolution due to its high surface area. The results showed that the standard UCC cleaning procedure as specified was acceptable.

1.1.3.4   Process Equipment — Vendor Designed

This activity included the engineering effort associated with specifying and selecting process equipment such as compressors, pumps, and tanks that will be designed and fabricated by commercial suppliers.

Equipment cleaning procedure "SCS-1" was issued. This procedure, specific to fabricated equipment for the silane portion of the process (e.g. silane column, condenser, storage tanks), was used by equipment suppliers as a final fabrication specification. For chlorosilane service up to the silane column, an existing specification (GS-40) was modified to cover all relevant equipment. Items which were vendor designed and fabricated include:

Item No.	Description
424-04	Reboiler
426-02	Quench Contractor Pump
426-06, 07	STC Pump
427-02	TCS Filter
429-02	Quench/Solids Removal Contactor
429-04	Gas Superheater Furnace
434-02	Stripper Condenser
434-06, 10	Reboilers
434-08, 14, 18	Column Condensers
434-12, 16, 24	Coolers
436-02	TCS Distillate Pump
436-04	DCS Distillate Pump
436-05	Spare Chlorosilane Pump
437-02	Silane Filter
444-02	Coolers

Item No.	Description
452-02	Tailing Column
453-02	Agglomerator Blower
453-04, 05	Waste Gas Blowers
456-08	Tailing Column Pump
459-02, 04	Venturi's
459-16, 20	Tank Agitators
417-02	M-G Silicon Unloading Filter
428-04	M-G Silicon Conveyor
454-04	Silica Agglomerator
457-04	Combustibles Vent Filter
457-06	Silica Bag Filter
459-08, 10, 12, 14	Waste Burner #1, #2, #3, #4
459-18	Caustic Tank, Heater
461-02	Hot Oil Tank
451-18	Caustic Storage Tank
461-04	Fuel Oil Day Tank

For a complete listing of equipment items, see Appendix A.

1.1.3.5 Auxiliary Equipment

This included the engineering activities associated with specifying and selecting auxiliary equipment such as heating and refrigeration systems, instrument air unit, and cooling tower which was designed and fabricated by commercial suppliers. Equipment items purchased under this classification included:

Item No.	Description
461-04	Fuel Oil Tank
466-06, 07	Fuel Oil Pumps
469-06	Cooling Tower Treatment
469-12	Refrigeration System
469-20	Instrument Air Dryer

Item No.	Description
643-02	Emergency Generator
469-02	Cooling Tower
459-18	Therminol Heater

Hydrogen and argon liquid storage tanks will be leased and thus are not re-quired as capital expenditure items.

1.1.3.6   Support Equipment

This effort included the engineering activities associated with specifying and selecting commercially-supplied equipment such as trailers and materials-handling systems.

Quality Control Laboratory

The modular laboratory is a unit 56 feet long x 12 feet wide equipped with appropriate work surfaces, cabinets, fume hoods, and electrical and plumbing fixtures. The sample line termination panel and analytical equipment were purchased separately. All components will be shipped to the UCC East Chicago facility for assembly and checkout prior to final connec-tions at the adjacent EPSDU site.

A layout of the sample line termination panel was developed. The panel face, 5 feet high x 6 feet wide, will allow display of sample flow, pressure, and manual sample-stream selection for the 47 process sample points. The panel was designed to be maintained at $165^{\circ}F$ to assure that all samples remain in the vapor phase for chromatographic analysis. Special considera-tion was given to safety and environmental concerns. A unique panel face ventilation arrangement was designed to vent any small leaks directly out-doors, and sandwich-type-construction insulation keeps the panel face cool while maintaining $165^{\circ}F$ inside.

The Q.C. laboratory included an UV spectrophotometer, a gas chromatograph system, a silicon melting furnace, and a plasma spectrometer.

The Jarrell-Ash inductively coupled plasma emission spectrometer was evaluated in detail. The functional need at EPSDU is for a multi-element determination of selected metallic elements in various chlorosilane, silane, and silicon samples. The concentration of these elements varies from 1000 ppm in metallurgical silicon and the waste metals stream to 1 ppb in the silane and silicon products. The unit should be relatively simple to operate on a routine basis and should require a minimum of analytical manpower for sample preparation and analysis. As a result of the evaluation of atomic absorption spectrometers (both flame and graphite furnace types), atomic absorption instruments with Zeeman optics, and induction-coupled argon plasma emission spectrometers, the plasma spectrometer was selected as the best unit for EPSDU. The Zeeman spectrometer appears to be potentially superior for detecting certain elements in high-purity silicon because of a unique background correction and large sample-size capability. However, the extremely non-linear response, the difficulty of loading precise, reproducible amounts of sample into the furnace, and the lack of in-house experience were negative aspects. All of the units evaluated could utilize the same sample preparation techniques. The Zeeman spectrometer can also accept a solid sample. The plasma spectrometer was selected because the detection and quantization limits are at least as good as the atomic absorption units, it provides simultaneous output of all selected elements, and it requires less trial-and-error dilution of samples and standards because of the broad linear response.

Trailer requirements were reviewed based on firm information from the analytical equipment vendors as to dimensions and services required. A single clean-air type acid fume hood was chosen to replace two conventional hoods and eliminate the need for filtering all laboratory ventilation to HEPA standards.

The Q.C. trailer specifications were revised to lower the trailer costs. The revised bid package resulted in a 50% reduction in unit cost without loss of function.

1.1.3.7   Other Equipment

The silane free-space reactor and the melting/consolidation system can be classified as "other equipment" and are described under the supporting R&D classification in tasks 1.6.1 and 1.62 respectively.

Items classified as "specialty items" are those items not readily available and were designed and fabricated/assembled at the Linde factory and include the following:

Chlorosilane Pump Pressure-Seal Systems

These assemblies provide for flushing a high-pressure fluid through the double mechanical seals of the centrifugal pump. One assembly is required per pump. This design was tried at Sistersville and appears to be the best way to seal the rotating shaft of a centrifugal pump in chlorosilane service. Each assembly consists of a small pneumatically-driven pump mounted on a 2-gallon container, together with appropriate local instrumentation such as level and pressure indicators. The components, ordered from sources recommended by Sistersville, were assembled at Tonawanda.

Hydrogenation Reactor Ram Valve

This valve isolates the metallurgical silicon feed from the hydrogenation reactor and consists of a pneumatically-driven air cylinder operator, a high pressure nitrogen-purged stuffing box seal, a therminol-jacketed stainless steel body, and an Incoloy 800 plug. The plug is attached to the air cylinder through a set of shafts, shaft seals, and the stuffing box. This valve assembly isolates the hot, corrosive environment of the hydrogenation reactor from the mild-steel equipment that feeds the metallurgical silicon. Since the Incoloy plug exactly fits the hydrogenation reactor feed port, there is no "dead space" for precipitation of the metal chloride vapors. The components were assembled at Tonawanda for shipment to the field.

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Mechanical Cleaner for the Agglomerator

A spring-type cleaner was chosen for the vertical tubes. These loosely-wound springs are friction-hung from the top feed-thru fitting of each agglomerator tube. Silica deposits will be scraped off by a vertical motion of the center which is hand-stroked. The construction material is high-tensile steel piano wire.

Stack Gas Analyzer/Leak Detector

The leak detector for the gas superheater is an instrument which continuously samples the superheater flue gas and sets off an alarm if a high level of HCl is detected. The detection method is to dissolve the stack gas in de-ionized water and test and solution with a chloride-sensitive electrode.

A commercial instrument was not available; a discontinued Beckman design was tailored to the exact needs. The analyzer, contained in a metal box, draws a sample of the stack gas through a magnetically stirred, constant temperature bath containing de-ionized water. A chloride ion probe senses any hydrogen chloride present in the stack gas, sending a signal to the pH meter which has a meter relay alarm feature.

Pyrolysis Hydrogen Compressor

The compressor was refitted and cleaned to meet electronic-grade purity specifications.

Sample Conditioners

The sample conditioners were designed, assembled and cleaned to electronic-grade purity specifications. These pipeline units adjust pressure and temperature of small sample streams that are directly piped to the Q.C. lab.

1.1.3.8   Data Collection System

The computerized data collection system for the 100 MT/Yr silane-to-silicon EPSDU plant provides operational guidance and design data for the commercial facility. The basis of this system is the computer control system which is used by UCC-Linde in the control of Air Separation Plants.

The main data-acquisition computer was delivered to Tonawanda for checkout prior to re-shipment to the EPSDU site.

The system selected was the Data General minicomputer and several input/output devices. Figure 1.18 shows a block diagram of the data collection system. The purpose of the data collection system is to:

- Provide data for the commercial design

- Provide operational guidance at EPSDU

The following paragraphs describe how this is accomplished:

Data to be Collected

The specific data which will be collected have been chosen so that heat and mass balances can be obtained, kinetic modeling can be accomplished, and the efficiencies of the heat exchanger and distillation columns can be determined. The remaining data points will be used to monitor control systems and provide operational guidance. The data can be separated into four groups according to how they are collected. These are:

- Temperatures, pressures, flow rates & levels
- Relay and switch operation
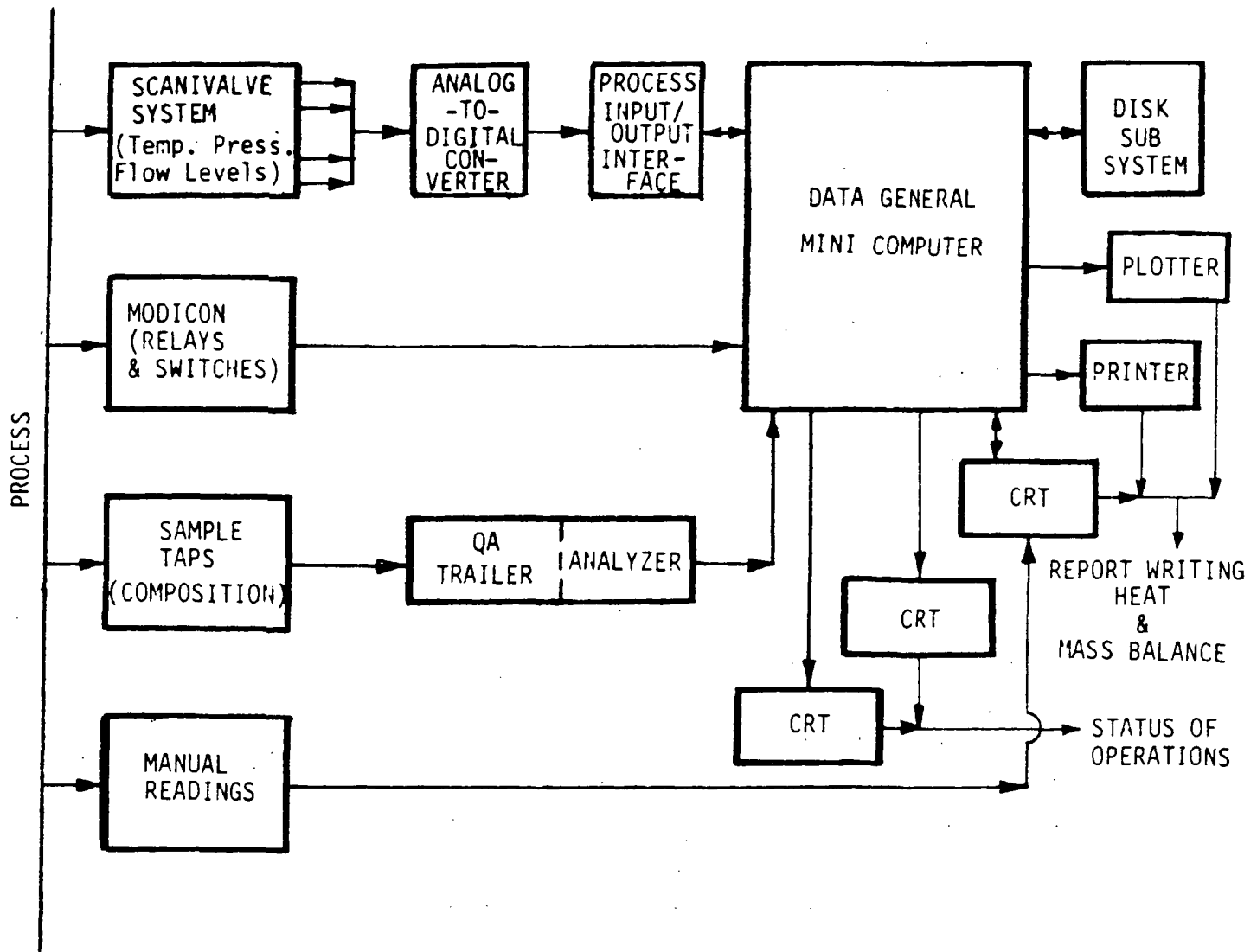- Composition
- Raw material and utilities usage

FIGURE 1.18   Data Collection Scheme

Method of Data Collection

Temperatures, pressures, flow rates, and levels are monitored using a Scanivalve System which consists of four modules and one drive shaft. The drive shaft is driven and the position of Scanivalve System is monitored by signals to and from the minicomputer. The signals (measurements) from the process are channeled through an analog-to-digital converter, through a process input/output interface, and finally into the minicomputer buffer. This information is converted to predesignated engineering units, assigned a label (location identification) and time, and stored in the memory.

The Modicon is filled with a self-contained programmable processor. This unit scans the on-off signals of the relays and switches. In the event of a change in the position of a switch, the information is sent to the minicomputer and is labeled with a location and a time.

Stream sampling and compositional determination will be provided by the quality analysis part of the EPSDU program. The interface between the Q.C. lab and the minicomputer will be a terminal which is also used as an output device for the gas chromatograph system. Analyses results will be typed in on the terminal with a compositional profile, a location label, and the time at which the sample was taken. This information will go the minicomputer and be stored in the memory bank.

Raw materials and utilities usage information will be based on a manual process involving the reading of gauges, etc. This information will be entered through a CRT (Cathrode Ray Tube) with a three element label: time, location, and quantity.

Method of Data Output

There will be five output devices — three CRT's, an X-Y plotter, and a serial printer. In addition, there is a disk subsystem.

One CRT will be in the control room and will be used by the operating
staff to monitor the status of the process. The remaining output devices
will be in the trailer. One CRT, the X-Y plotter and the serial printer
will be used for report generation, operations, planning, and examination
of process variables. The other CRT will operate in the same mode as the
control room CRT and will be used for trouble-shooting problem areas such
as control systems. The disk subsystem will be used to generate data
disk packs which will be sent to Tonawanda for data reduction.

Form of Output

There will be two forms of formal outputs. The first will be
an approximately weekly operations report that includes a mass balance,
heat balance, comments on operations, and various other pertinent data
for that time period. The other formal output will be the results of
the data reduction at Tonawanda.

Software Requirements

The software requirements can be separated into two areas:

- Data Acquisition and Storage Programming
- Data Reduction Programming

The first area was handled by the Linde Tonawanda minicomputer
group. This work included the implementation of the existing software
system for data acquisition, conversion, and trending as well as the
development of any new software required for data storage and retrieval.
The second area was the responsibility of the Process R&D group — this
area involved two phases: one at EPSDU and one at Tonawanda. The EPSDU
phase includes:

- The first part of the programming will involve making
  the data readily available in a usable form. This
  will require a data sorting program, a plotting program,
  an integration program and a physical properties package

- The second part is assembling the information for heat and mass balances and performing these balances.

- The third part is the formatting required for report generation, input from the Q.C. lab, and input from manual readings.

The Tonawanda phase includes:

- Preliminary treatment of data such as sorting and integrating

- Hydrogenation kinetics

- TCS redistribution kinetics

- DCS redistribution kinetics

- Vapor-Liquid Equilibrium data, column efficiencies

- Heat exchanger heat transfer coefficients

- Pyrolysis and waste treatment parameters

Software for the data collection system was prepared jointly by process engineers and Computer Applications personnel. Software already developed was modified as were routines necessary to enter the data into the minicomputer memory. The process group provided the computer group with necessary information such as a parameter list, parameter ranges, and the frequency of collection. Additionally, the process group prepared software to output the data in formatted reports.

The Nova IV minicomputer, 2 CRTS, a line printer, twenty 5-megabyte disk packs, and 10 double-density floppy disks (diskettes) were received and installed at Tonawanda. The 5-megabyte disk packs and the floppy disks have been initialized and formatted. This procedure established the record length, record density, and labeling to permit random access of data stored in the disks and diskettes. The software provided by Data General and standard software developed by UCC was loaded into the permanent memory and includes utility programs and library files.

Installed in the same area as the peripherals were a Hewlett-Packard gas chromatograph electronic controller and remote input/output (I/O) station. The Hewlett-Packard interface components were installed and tested to ensure proper interfacing between the controller and the Data General minicomputer.

Process Variable List

The process variable list was completed and stored on a disk pack in the minicomputer. Programs which allow the listing, sorting and the alteration of the list were also transferred onto this disk pack. The process variable list included an internal identification number, an external identification number, a description, engineering units, signal type, signal location (Panel Number), an instrument tag, and the variable class (see Table IV). The two identification numbers are the minicomputer's primary means of searching for a specific variable. The description is the user's primary means of searching for a specific variable. The units are the storage locations of the process variable value. Signal type is the type of signal which is sent from the instrumentation:

PN = pneumatic (psig)

T/C = thermocouple (mvolts)

E = electronic (mamps)

The panel locations have not yet been assigned. The instrument tag is the identification number of the instrument from which the signal originates and the variable class identifies the type of Scanivalve system from which the signal is collected; 3 pneumatic, 4 electronic, and 5 thermocouple.

The process I/O system is the interface between the process equipment (thermocouples, flow meters, level indicators) and the minicomputer. Most of the signals (184 signals) will be routed through a scanivalve system which has four modules — one thermocouple, one electronic, and two pneumatic. Signals from the modules travel through four channels

TABLE IV

SILICON CPSDU PROCESS VARIABLE LIST

11/11/80

ID		ED	DESCRIPTION	UNITS	SIG	PANEL	INST.TAG	CLASS	COMMENT
PV	1	ABT071	MAKE-UP H2 TO HYD REA TEMP	DEG F	E	–	TE107-1	4	S RTD
PV	2	ABT072	HYDROGENATION REA TEMP #1	DEG F	E	–	TE107-2	4	S RTD
PV	3	CDP172	MAKE-UP H2 TO HYD REA PRES	PSIG	PN	–	PT317-2	3	
PV	4	ABT231	SUPHEA STC TO HYD REA TEMP	DEG F	E	–	TE123-1	4	S RTD
PV	5	ABF281	STC FLOW FROM STC STORAGE	ACFM	PN	–	FIT128-1	3	WALL & TIER
PV	6	ABT221	SUPHEAT H2 TO HYD REA TEMP	DEG F	E	–	TE122-1	4	S RTD
PV	7	ABF211	COMPRSD H2 FLOW TO SH FURN	ACFM	PN	–	FT121-1	3	WALL & TIER
PV	8	ABT073	HYDROGENATION REA TEMP #2	DEG F	E	–	TE107-3	4	S RTD
PV	9	ABT074	HYDROGENATION REA TEMP #3	DEG F	PN	–	TT107-1	3	
PV	10	ABL071	HYDROGENATION REA LEVEL	PSIG	PN	–	LT107-1	3	D/P CELL
PV	11	ABD072	HYDROGENATION DELTA P	PSIG	PN	–	PDT107-2	3	D/P CELL
PV	12	ABP071	HYDROGENATION INLET PRESS	PSIG	PN	–	PT107-1	3	
PV	13	ABT081	HYDROGENATION OUTLET TEMP	DEG F	E	–	TE108-1	4	S RTD
PV	14	ACT082	WASTE SETTLER TANK TEMP	DEG F	T/C	–	TE108-2	5	TYPE T
PV	15	ACT111	RECYCLE TO QC VENTURI TEMP	DEG F	T/C	–	TE111-1	5	TYPE T
PV	16	ACF171	QUENCH CONDENSER WASH FLOW	ACFM	PN	–	FIT117-1	3	WALL & TIER
PV	17	ACD132	QUENCH CONDENSER FEED DP	PSIG	PN	–	FT113-2	3	D/P CELL
PV	18	ACT141	QUENCH RECEIVER TEMP	DEG F	T/C	–	TE114-1	5	TYPE T
PV	19	ACF191	CRUDE TCS FLOW TO STORAGE	ACFM	PN	–	FIT119-1	3	WALL & TIER
PV	20	ACY161	VALVE POS ON RECYLCE TO QC	% OPEN	PN	–	–	3	NOT SPEC
PV	21	ACP181	H2 PRESS IN RECYCLE RECEIV	PSIG	PN	–	PT118-1	3	
PV	22	ACT181	H2 TEMP IN RECYCLE RECEIV	DEG F	T/C	–	TE118-1	5	TYPE T
PV	23	ACL251	TCS STORAGE TANK LEVEL	FEET	–	–	LT125-1	0	DREX/DIRECT
PV	24	ABT241	SAT. STC FROM VAPORIZOR T	DEG F	T/C	–	TE124-1	5	TYPE T
PV	25	ABT075	HYDROG REA STC FEED TEMP	DEG F	E	–	TE107-5	4	S RTD
PV	26	AAL001	LEVEL OF M.G. SILICON BIN	FEET	–	–	LI100-1	0	DIRECT
PV	27	BAF251	STRIPPER COLUMN FEED FLOW	ACFM	PN	–	FIT125-1	3	WALL & TIER
PV	28	BAT257	STRIPPER COLUMN FEED TEMP	DEG F	T/C	–	TE125-7	5	TYPE T
PV	29	BAT251	STRIPPER COLUMN TEMP #1	DEG F	T/C	–	TE125-1	5	TYPE T
PV	30	BAT252	STRIPPER COLUMN TEMP #2	DEG F	T/C	–	TE125-2	5	TYPE T
PV	31	BAT253	STRIPPER COLUMN TEMP #3	DEG F	T/C	–	TE125-3	5	TYPE T
PV	32	BAT254	STRIPPER COLUMN TEMP #4	DEG F	T/C	–	TE125-4	5	TYPE T
PV	33	BAT255	STRIPPER COLUMN TEMP #5	DEG F	T/C	–	TE125-5	5	TYPE T
PV	34	BAT256	STRIPPER COLUMN TEMP #6	DEG F	T/C	–	TE125-6	5	TYPE T
PV	35	BAT031	STRIPPER OVHD TEMP	DEG F	T/C	–	TE203-1	5	TYPE T
PV	36	BAT041	STRIPPER CONDENS LIQD TEMP	DEG F	T/C	–	TE204-1	5	TYPE T
PV	37	BAT501	STRIPPER CONDENS VAPR TEMP	DEG F	T/C	–	TE2501-1	5	TYPE T
PV	39	BAF501	STRIPPER CONDENS VAPR FLOW	ACFM	PN	–	FT2501-1	3	WALL & TIER
PV	40	BAD041	STRIPPER REFLUX DP	PSIG	PN	–	FT204-1	3	ORIFICE

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on an analog-to-digital (A/D) card and a digital signal enters the computer. There are approximately 20 analog signals which do not go through the scanivalve system but are directly associated with a dedicated channel on an A/D card. There is no economical balance between the cost of the channels and the cost of an additional scanivalve module; the system described above has been chosen as being the most efficient and reliable.

The digital signals which enter the computer are converted into values in engineering units (deg F, psig, ACFM, etc.) which are then stored on the disk pack.

A system for data reduction operation at Tonawanda has been identified. The full disk packs of data obtained at EPSDU will be sent to Tonawanda where the data will be transferred to magnetic type for reading into the UCC main-frame computer network. A combination of sorting and statistical routines will be used to reduce the data to a usable form. The data will then be entered into several project-specific engineering programs which will produce operations information and design parameters.

On-line data (scanivalve points and direct inputs) are collected at specified time intervals by the computer. The off-line data (analyses and manual readings) are manually entered through one of the peripheral devices. Information about the process can be obtained in either of two ways:

(1) Automatic readout in the form of a log, a daily report, or a message,

or

(2) Operating personnel can request by typing manual instructions to the computer to display a group of process variables or an hourly average of key variables for the past 24-hour period.

Daily Report

The daily report summarizes the key performance parameters viz:

1. Specified primary process variables (approx. 50) vs clock
   time; one hour averages, daily averages and deviations

   - Flows on ACFM or GPM
   - Temperatures in $^{\circ}F$
   - Pressures in psia
   - Levels in inches

2. Heat and Mass Balance

   a. Overall facility Si/H/Cl balance

   b. Sub-system balance

   - Hydrogenation reactor/superheater
   - Quench condenser loop
   - Hydrogenation sub-system
   - Stripper Column
   - TCS Column/TCS Redistribution Reactor
   - DCS Column
   - Silane Column/DCS Redistribution Reactor
   - Distillation sub-system
   - Pyrolysis/Consolidation sub-system

   c. Minimum inclusion into daily report will be based
      on daily averages, but hourly averages can also be
      be used as required.

3. Utility consumptions, absolute and pro-rata

   - Electric power
   - Natural gas
   - Water
   - Nitrogen
   - Argon

4. Off-line/waste treatment inputs

- Q.C. trailer off-line analyses
- Process off-line inputs
- Waste treatment area flows, differential pressures, and temperatures

A sample of the daily report (4 pages) is shown in Table V.

1.1.4 INSTALLATION DESIGN, SPECIFICATION, SUBCONTRACT

This design effort includes development of separate installation drawing packages for the site, civil, mechanical, and electrical specialties based on the engineering design effort and vendor-supplied information. Specification activity includes definition of specific requirements for performing all installation functions. Subcontracting includes the preparation of bid packages, evaluation of quotes, subcontractor selection and contract negotiation.

1.1.4.1 Site Drawing & Specs

This work item included preparation of all drawings and specifications necessary for rough grading, establishing drainage patterns, installing fencing, providing a base for parking lots and roads.

The Site Preparation Package was awarded to Vic Kirsch Construction Co., Inc., 7020 Cline Avenue, Hammond, Indiana 46223, on June 18, 1980. The package contained the following drawings:

D-2146801 - General Plan
D-2146802 - Location Plan
D-2146803 - Details and Specifications

Site Preparation work, as defined in the Site Preparation Package, consisted of the following:

TABLE V - DAILY OPERATIONS REPORT (PAGE 1 of 4)

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EXPERIMENTAL PROCESS DEVELOPMENT UNIT

SILANE-TO-SILICON PROCESS

DAILY OPERATIONS REPORT
DATE: 7 6 81
TIME: 8 33 18

**   OVERALL MASS FLOW - SILANE FACILITY   **

CONSUMED(LB/HR)	PRODUCT(LB/HR)	WASTE(LB/HR)	RECYCLE(LB/HR)
H2 = 1000.0	SIH4= 1000.0	H2 = 1000.0	DCS = 1000.0
TCS = 1000.0		SIH4= 1000.0	TCS = 1000.0
STC = 1000.0		HCL = 1000.0	STC = 1000.0
SI = 1000.0		MCS = 1000.0	
CU = 1000.0		DCS = 1000.0	
N2 = 1000.0		TCS = 1000.0	
		STC = 1000.0	
		SI = 1000.0	
		CU = 1000.0	
		N2 = 1000.0	

**   HYDROGENATION MASS FLOWS   **

CONSUMED(LB/HR)	PRODUCT(LB/HR)	WASTE(LB/HR)	RECYCLE(LB/HR)
H2 = 1000.0	H2 = 1000.0	H2 = 1000.0	H2 = 1000.0
HCL = 1000.0	HCL = 1000.0	HCL = 1000.0	HCL = 1000.0
DCS = 1000.0	MCS = 1000.0	DCS = 1000.0	DCS = 1000.0
TCS = 1000.0	DCS = 1000.0	TCS = 1000.0	TCS = 1000.0
STC = 1000.0	TCS = 1000.0	STC = 1000.0	STC = 1000.0
SI = 1000.0	STC = 1000.0	SI = 1000.0	N2 = 1000.0
CU = 1000.0	N2 = 1000.0	CU = 1000.0	
N2 = 1000.0		N2 = 1000.0	

**   DISTILLATION MASS FLOWS   **

CONSUMED(LB/HR)	PRODUCT(LB/HR)	WASTE(LB/HR)	RECYCLE(LB/HR)
H2 = 1000.0	SIH4= 1000.0	H2 = 1000.0	DCS = 1000.0
HCL = 1000.0		SIH4= 1000.0	TCS = 1000.0
MCS = 1000.0		HCL = 1000.0	STC = 1000.0
DCS = 1000.0		MCS = 1000.0	
TCS = 1000.0		DCS = 1000.0	
STC = 1000.0		N2 = 1000.0	
N2 = 1000.0			

**   PYROLYSIS/MELTING MASS FLOWS   **

CONSUMED(LB/HR)	PRODUCT(LB/HR)	WASTE(LB/HR)	RECYCLE(LB/HR)
SIH4= 1000.0	SI = 1000.0	H2 = 1000.0	H2 = 1000.0
		SIH4= 1000.0	
		SI = 1000.0	

```
*
**
***
****                            UTILITIES/RAW MATERIALS REPORT


**                   UTILITIES USAGE                    **

    C WATER   =  1000.0 GPM       OUT =  1000.0 DEG F
                                  RET =  1000.0 DEG F
    THERMINOL =  1000.0 GPM       HOT =  1000.0 DEG F
                                  RET =  1000.0 DEG F
    REFRIGERNT=  1000.0 GPM       OUT =  1000.0 DEG F
                                  RET =  1000.0 DEG F


    NAT GAS   =  1000.0 ACF    DATE: 10 10 TO 10 10
    ELECTRICAL=  1000.0 KWH    DATE: 10 10 TO 10 10
    NITROGEN  =  1000.0 ACF    DATE: 10 10 TO 10 10


**              RAW MATERIALS- ON HAND                  **

        STC TANK  =  1000.0 GAL
        MG SI BIN =  1000.0 LBS
        DRIOX H2  =  1000.0 ACF
*
**
***
****                           HYDROGENATION DETAILED REPORT


**              ATOMIC MASS FLOW        **              **              HEAT DUTY REPORT       **

    LBS    IN            LBS    OUT          HT EXCHANGER DUTY BTU/HR

    H   =  1000.0        H   =  1000.0       VAPORIZOR  =  1000.0
    CL  =  1000.0        CL  =  1000.0       SUPERHEATR =  1000.0
    SI  =  1000.0        SI  =  1000.0       Q CONDENSR =  1000.0


**                  REACTOR               **              **              W SETTLER             **

        TEMP      =  1000.0 DEG F               TEMP       =  1000.0 DEG F
        PRESSURE  =  1000.0 PSIA                PRESSURE   =  1000.0 PSIA
        % CU      =  1000.0                     RECYCLE TO =  1000.0 LBS/HR
        H2:STC    =  1000.0                     RECYCLE FR =  1000.0 LBS/HR
        CONVERSION=  1000.0 LBS                 DUMPED     =  1000.0 LBS
        PART SIZE =  1000.0 MICROM


**                  Q CONDENSR            **              **              TCS TANK              **

        DELTA P   =  1000.0 PSI                 LEVEL    =  1000.0 INCHES
        WASH FLOW =  1000.0 LBS/HR              DELTA P  =  1000.0 PSI
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DISTILLATION AREA DETAILED REPORT

** STRIPPER COLUMN ** ** TCS COLUMN **

TOTAL FEED (LBS/HR)	DISTILLATE (LBS/HR)	BOTTOMS (LBS/HR)	TOTAL FEED (LBS/HR)	DISTILLATE (LBS/HR)	BOTTOMS (LBS/HR)
H2 = 1000.0	H2 = 1000.0	H2 = 1000.0	SIH4= 1000.0	SIH4= 1000.0	SIH4= 1000.0
SIH4= 1000.0	SIH4= 1000.0	SIH4= 1000.0	HCL = 1000.0	HCL = 1000.0	HCL = 1000.0
HCL = 1000.0	HCL = 1000.0	HCL = 1000.0	MCS = 1000.0	MCS = 1000.0	MCS = 1000.0
MCS = 1000.0	MCS = 1000.0	MCS = 1000.0	DCS = 1000.0	DCS = 1000.0	DCS = 1000.0
DCS = 1000.0	DCS = 1000.0	DCS = 1000.0	TCS = 1000.0	TCS = 1000.0	TCS = 1000.0
TCS = 1000.0	TCS = 1000.0	TCS = 1000.0	STC = 1000.0	STC = 1000.0	STC = 1000.0
STC = 1000.0	STC = 1000.0	STC = 1000.0			
N2 = 1000.0	N2 = 1000.0	N2 = 1000.0			

REFLUX RAT= 1000.0		PRESSURE = 1000.0 PSIA	REFLUX RAT= 1000.0		PRESSURE = 1000.0 PSIA
CONDENSER = 1000.0 BTU/HR		TEMP = 1000.0 DEG F	CONDENSER = 1000.0 BTU/HR		TEMP = 1000.0 DEG F
REBOILER = 1000.0 BTU/HR		TEMP = 1000.0 DEG F	REBOILER = 1000.0 BTU/HR		TEMP = 1000.0 DEG F
SEPARATION= 1000.0		TEMP = 1000.0 DEG F	SEPARATION= 1000.0		TEMP = 1000.0 DEG F

** DCS COLUMN ** ** SIH4 COLUMN **

TOTAL FEED (LBS/HR)	DISTILLATE (LBS/HR)	BOTTOMS (LBS/HR)	TOTAL FEED (LBS/HR)	DISTILLATE (LBS/HR)	BOTTOMS (LBS/HR)
SIH4= 1000.0	SIH4= 1000.0	SIH4= 1000.0	SIH4= 1000.0	SIH4= 1000.0	SIH4= 1000.0
HCL = 1000.0	HCL = 1000.0	HCL = 1000.0	HCL = 1000.0	HCL = 1000.0	HCL = 1000.0
MCS = 1000.0	MCS = 1000.0	MCS = 1000.0	MCS = 1000.0	MCS = 1000.0	MCS = 1000.0
DCS = 1000.0	DCS = 1000.0	DCS = 1000.0	DCS = 1000.0	DCS = 1000.0	DCS = 1000.0
TCS = 1000.0	TCS = 1000.0	TCS = 1000.0	TCS = 1000.0	TCS = 1000.0	TCS = 1000.0
STC = 1000.0	STC = 1000.0	STC = 1000.0	STC = 1000.0	STC = 1000.0	STC = 1000.0

REFLUX RAT= 1000.0		PRESSURE = 1000.0 PSIA	REFLUX RAT= 1000.0		PRESSURE = 1000.0 PSIA
CONDENSER = 1000.0 BTU/HR		TEMP = 1000.0 DEG F	CONDENSER = 1000.0 BTU/HR		TEMP = 1000.0 DEG F
REBOILER = 1000.0 BTU/HR		TEMP = 1000.0 DEG F	REBOILER = 1000.0 BTU/HR		TEMP = 1000.0 DEG F
SEPARATION= 1000.0		TEMP = 1000.0 DEG F	SEPARATION= 1000.0		TEMP = 1000.0 DEG F

** TCS REDISTRIBUTION REACTOR ** ** DCS REDISTRIBUTION REACTOR **

LBS/HR IN	LBS/HR OUT	LBS/HR IN	LBS/HR OUT
SIH4= 1000.0	SIH4= 1000.0	SIH4= 1000.0	SIH4= 1000.0
HCL = 1000.0	HCL = 1000.0	HCL = 1000.0	HCL = 1000.0
MCS = 1000.0	MCS = 1000.0	MCS = 1000.0	MCS = 1000.0
DCS = 1000.0	DCS = 1000.0	DCS = 1000.0	DCS = 1000.0
TCS = 1000.0	TCS = 1000.0	TCS = 1000.0	TCS = 1000.0
STC = 1000.0	STC = 1000.0	STC = 1000.0	STC = 1000.0

TOTAL = 1000.0 LBS/HR		TOTAL = 1000.0 LBS/HR	
TEMP = 1000.0 DEG F		TEMP = 1000.0 DEG F	
PRESSURE = 1000.0 PSIA		PRESSURE = 1000.0 PSIA	
CL:H = 1000.0		CL:H = 1000.0	
CONVERSION= 1000.0 LBS		CONVERSION= 1000.0 LBS	

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*
**
***
****                              PYROLYSIS/MELTING DETAILED REPORT


**                    REACTOR              **          **                      MELTERS            **


      LBS   IN              LBS   OUT                      LBS   #1              LBS   #2

      SIH4=  1000.0         SIH4=  1000.0                  SI  = 1000.0         SI  = 1000.0
                            H2  =  1000.0
                            SI  =  1000.0

      TEMP      = 1000.0 DEG F                   TEMP      #1 = 1000.0 DEG F TEMP      #2 = 1000.0 DEG F
      PRESSURE  = 1000.0 PSIA                    PRESSURE  #1 = 1000.0 PSIA  PRESSURE  #2 = 1000.0 PSIA
      ELECTRICAL= 1000.0 KW                      ELECTRICAL#1 = 1000.0 KW    ELECTRICAL#2 = 1000.0 KW
      ARGON     = 1000.0 ACF                     ARGON     #1 = 1000.0 ACF   ARGON     #2 = 1000.0 ACF
*
**
***
****                          WASTE TREATMENT AREA DETAILED REPORT


**              WASTE BURNER #1                **    **              WASTE BURNER #2                **

      THROUGHPUT= 1000.0 LBS                         THROUGHPUT= 1000.0 LBS
      TEMP      = 1000.0 DEG F                        TEMP      = 1000.0 DEG F
      COMB AIR  = 1000.0 ACF                          COMB AIR  = 1000.0 ACF
      NAT GAS   = 1000.0 ACF                          NAT GAS   = 1000.0 ACF

**              WASTE BURNER #3                **    **              LIQUID BURNER                 **

      THROUGHPUT= 1000.0 LBS                         THROUGHPUT= 1000.0 LBS
      TEMP      = 1000.0 DEG F                        TEMP      = 1000.0 DEG F
      COMB AIR  = 1000.0 ACF                          COMB AIR  = 1000.0 ACF
      NAT GAS   = 1000.0 ACF                          NAT GAS   = 1000.0 ACF
```

- Remove all bricks, railroad ties, tires, oil drums, concrete, metal sheets, pipes, shrubs and trees within the property fence and place in existing oil pond east of fence line.

- Strip the 0 - 12 inches of top soil and grub any root systems within the outer shoulder of the roadway loop and place in oil pond.

- Grade site as indicated on drawings. Place excess material in existing oil pond and rough grade.

- Strip roadway and parking areas a minimum of 9 inches and proof-roll. Back-fill with a minimum of 9 inches of granular base, as specified, and compact to a minimum of 95% of maximum dry density.

- Clean and clear existing boundary fence and repair as required.

- Install permanent and temporary fencing and three new gates.

- Install roadway guardrail.

- Install three permanent construction monuments as located on drawings.

1.1.4.2   **Civil Design, Specifications and Materials**
and
1.1.4.3   **Civil Drawing**

This activity included preparation of specifications and material requirements necessary for constructing foundations, shelters, pipe racks, structures, drainage ditches, underground utilities, and fire-water systems.

The civil installation design package included the following EPSDU installation items:

- General Plot Plan
- Site Preparation (Reference)

- Foundation/Caisson Location
- Equipment Foundations Details
- Structure Foundation Details
- Underground Piping and Utilities
- Underground Tankage and Basins
- Switchgear/Control Room Framing
- Melter Building Framing
- Gantry Structure Framing

The civil contract was awarded to the Edward Gray Corporation.

1.1.4.4   **Mechanical Design, Specifications and Materials**
and
1.1.4.5   **Mechanical Drawings**

These work items related to specifying the design requirements of the EPSDU and the preparation of drawings and specifications leading to a mechanical design package to be used for obtaining contractor bids for assembly of the mechanical items. The mechanical bid package for the silane section of the EPSDU contained:

- Set of mechanical drawings (65 mechanical and 43 instrumentation drawings).

- Field bill of materials which identifies each component in the EPSDU facility and indicates whether its mechanical installation rests with the subcontractor or UCC, Linde (several hundred pages). Although many of the piping components are standard, it was elected to purchase some of these items to insure proper type or because they will require special chlorosilane/silane cleaning at Tonawanda. Piping components and materials include valves, gaskets, filters, pressure and temperature indicators.

- Procedural specification for cleaning silane (stringent requirements) and chlorosilane (less stringent requirements) fabricated assemblies.

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- Specifications for piping and insulation for both the process and utility lines.

- Specifications for piping material for both process and utility lines and includes insulation, painting, and location of liquid drain lines.

- Procedural specifications for pressure testing and leak testing of chlorosilane and silane piping.

The bids for the EPSDU mechanical work were received and evaluated. A contract was not awarded since the EPSDU funding for construction was cut. Final drawing and specification changes were made and the mechanical installation package was marked "For Construction/Hold".

1.1.4.6   Electrical Design, Specification and Materials
and
1.1.4.7   Electrical Drawings

Electrical installation drawings were completed showing the location of underground conduit, yard lighting poles, and underground grounding cables. These drawings represent the initial electrical work, in addition to the power feeder, which was completed as part of the civil package.

An EPSDU site drawing that identifies all lighting requirements was prepared. In addition to the perimeter light standards and fixtures, lights will be mounted on the top corners of the gantry structure and the melter building to illuminate the central areas of the site. Lighting plans were developed for the interior of the gantry, melter building, and control room building.

Locating and labeling of all electrical process control instruments and devices on drawings proved to be an involved task due to the extensive process control/data collection system designed and specified for EPSDU. The location of each locally-mounted instrument was identified; cable-tray and conduit locations were identified and detailed drawings developed.

The bids for the EPSDU electrical work were received and evaluated. A contract was not awarded since the EPSDU funding for construction was cut. Final drawing and specification changes were completed and the electrical installation package was marked "For Construction/Hold".

1.1.5   COST ESTIMATING

1.1.5.1   Facility Cost Estimate

A detailed cost estimate was prepared for the EPSDU facility design, construction, and checkout. The costs were based on information generated during the engineering design and equipment specification activities of Phase II. The cost estimate was incorporated into the baseline budget and the performance measurements system (PMS) document.

1.1.5.2   Engineering Design Support

A "first-round" cost improvement review was conducted in April 1980 to identify the cost impact of various facility design changes which occured since "fixing" the design in December 1979. Cost reductions in the following areas were identified:

- M-G Silicon Storage/Preparation Conveyor - Hopper building eliminated plus conveyor system added.

- Hydrogenation Reactor - Redesigned to smaller size.

- Silane Column - Redesigned from tray column to smaller packed column.

- Gantry Structure - Reduced from five to three levels due to smaller silane column.

- Dryout System - Heater and piping added for initial "dryout" system (cost increase).

- Waste Treatment System - Waste neutralized and sent to sewer versus converting wastes to muriatic acid.

- **Spill Collection** - System redesigned to use underground pipe versus concrete troughs with grading.

- **Instrumentation** - Process control instrumentation re-estimated based on current P&I.

- **Electrical Power Supply** - Primary power from NIPSCO versus Linde facility.

1.1.6   PYROLYSIS/MELTING SYSTEM DESIGN

This task consisted of preparing an integrated design package for the pyrolysis/melting system sized for the EPSDU pilot plant. Information generated from the PDU free-space reactor experiments and the shotter/melter experimental work by Kayex (performed under a subcontract to Union Carbide) served as the basis for the resultant design.

Free-Space Reactor Hopper Design

The previously specified free-space reactor design was modified to incorporate new design features as follows:

- Replacement of the safety valve with a bursting disk.

- The vacuum line to the hopper was relocated to prevent plugging with powder.

- Mounting lugs replaced with flat pads.

- Power level detectors were added to the silicon powder hopper design.

- The powder transfer line from the hopper was respecified to use internal polished stainless steel tubing.

- The agitator shaft which contains breaker bars to prevent powder bridging has been relocated at a much lower level in the cone section of the hopper.

- To provide better aeration to the cone section, the porous cone was removed and replaced with a 24" diameter flat porous plate. This was done on the recommendation of personnel from the Polyolefins Division of UCC.

- A major modification was made in the design of the support and seal for the quartz liner in the free-space reactor. The previous design for the seal of the quartz liner to the metal reactor consisted of a 3/8" thick Grafoil ring shich would move about 1-1/2" when the reactor heats up to about 980°C. Because of this large movement and also that the Grafoil ring is friable, this design will not result in a reliable seal. The new design uses a sliding metal-to-metal contact and a metal bellows to provide a seal.

Melter Hopper and Excess Powder Hopper Design

The melter hopper is designed to receive powder transferred from the silicon powder hopper and provide storage of powder for the melting operation. When the silicon powder is not to be made into shot, the powder will transfer into the excess powder hopper where it will ultimately be disposed of by placing into 55 gallon drums.

Vessel Support Structure

A steel structure is necessary to support the free-space reactor/hopper, the melter/shotting system and the required vessels and material that are essential to the consolidation of silane into solid silicon. The structure envelope is 12 feet wide by 28 feet long and 31 feet high at the free-space reactor section, and 46 feet high in the melter/shotter section.

P&I Diagrams

Process and instrument diagrams were developed for the complete system. A P&I diagram is included for a fluid bed system which functions as a cushion to collect the shot falling from the melter.

Drawings

A drawing package of the complete system was generated and will serve as a basis for constructing and installing a suitable system in the EPSDU facility.

1.2   EQUIPMENT FABRICATION/DELIVERY

This report item included all in-house and outside activity associated with fabrication, delivery, and vendor coordination for all items of equipment. The report item was divided into the following tasks:

1.2.1   Process Control and Data Collection System
1.2.2   Electrical Power System
1.2.3   Process Equipment, Linde Designed
1.2.4   Process Equipment, Vendor Designed
1.2.5   Auxiliary Equipment
1.2.6   Support Equipment
1.2.7   Melting Equipment
1.2.8   Procurement Support

Each task is described and pertinent comments are made. It is assumed that the reader has each quarterly report available if he wishes to check monthly progress through the reporting period. A list of major equipment items purchased for the EPSDU facility has been developed to indicate tag number, component description, bill of material or specification number, and purchase order. This listing is included as Appendix A. Photographs of major equipment items and a report on condition received were recorded in an Inspection Log.

1.2.1   PROCESS CONTROL AND DATA COLLECTION SYSTEM

This task includes the purchase of field instruments and controls, panels and panel instruments and data collection system. Approximately 150 purchase orders were issued (for 1300 items).

1.2.2   ELECTRICAL POWER SYSTEM

Included in this task is the purchase of the power supply, power control center and other power system equipment.

1.2.3   PROCESS EQUIPMENT — LINDE DESIGNED

Four distillation columns were fabricated at UCC and seven reators were fabricated by vendors.

1.2.4   PROCESS EQUIPMENT — VENDOR DESIGNED

This task included the effort required for the fabrication, delivery and acceptance of compressors, pumps, heat exchangers, tanks, solids handling equipment, waste treatment equipment and other process equipment. A total of 41 purchase orders were issued (74 items).

1.2.5   AUXILIARY EQUIPMENT

Auxiliary equipment included the therminol heater, refrigeration unit, gas storage tanks and other associated equipment.

1.2.6   SUPPORT EQUIPMENT·

Support equipment was classified as the items associated with the purchase and fitting out of the quality control (Q.C.) trailer.

1.2.7   PROCUREMENT SUPPORT

This task includes the Procurement Department effort necessary to initiate, monitor and control the purchase of equipment. It also includes work associated with cleaning specialty items to strict standards, since the component suppliers cannot meet the specifications.

Approximately 600 items were cleaned to meet the chlorosilane service specification and an additional 150 items were cleaned to meet electronic-grade silane specifications. The cleaning took place at the Linde laboratories and the parts were packaged and shipped to the EPSDU site.

Shop visits were made to selected vendors to inspect major equipment items during fabrication to ensure conformance to specifications. A materials receiving log was maintained at the EPSDU site which gave details of equipment received, date of receipt and storage location on the site.

1.3 INSTALLATION AND CHECKOUT

This report item included all efforts associated with the award of subcontracts, monitoring subcontractor work and performance.

Initially a contract was awarded to Dames and Moore, Chicago, to make soil borings and soil analysis at the EPSDU East Chicago site. The site preparation was completed in August 1980 and included rough grading, fencing, parking lot and roadways. The site was adjacent to the Linde, East Chicago Gas Plant and the necessary gas supply could be easily tied-in. During early September, 1980, an installation contract was awarded to Edward Gray Corporation, Chicago, for all civil, structural, and underground utility work at EPSDU. (They submitted the lowest of six bids). The contractor moved on-site and began foundation work in late September. The operations personnel at East Chicago supervised the site preparation work and a construction superintendent (UCC employee) supervised the civil installation work.

All equipment and building foundations and all steel for the gantry was completed in the last quarter of 1980. The balance of the contractual work (Pyrolysis/Consolidation, Control and Waste Treatment buildings) was completed in the following quarter. Delivery of major equipment items began in the first quarter of 1981 and was complete in the second quarter. Off-loading, rigging, and setting equipment was effectively carried out by the Linde in-house maintenance contractor.

Purchase order for items better purchased by UCC than the contractor to ensure timely delivery and proper type (approximately 1900 special items or long lead quantity items) were issued. Figure 3.1, EPSDU Site, is a photograph of the site taken in March 1981 and shows construction progress. The on-site construction superintendent was supported by a clerk on a part-time basis whose responsibility was to maintain a "material receiving log" shown elsewhere in this report. Items with costs in excess of $1,000 were identified by government tags.

Figures 3.2 through 3.7 show equipment received at the site during April 1981 and were awaiting installation on their respective foundations and supports.
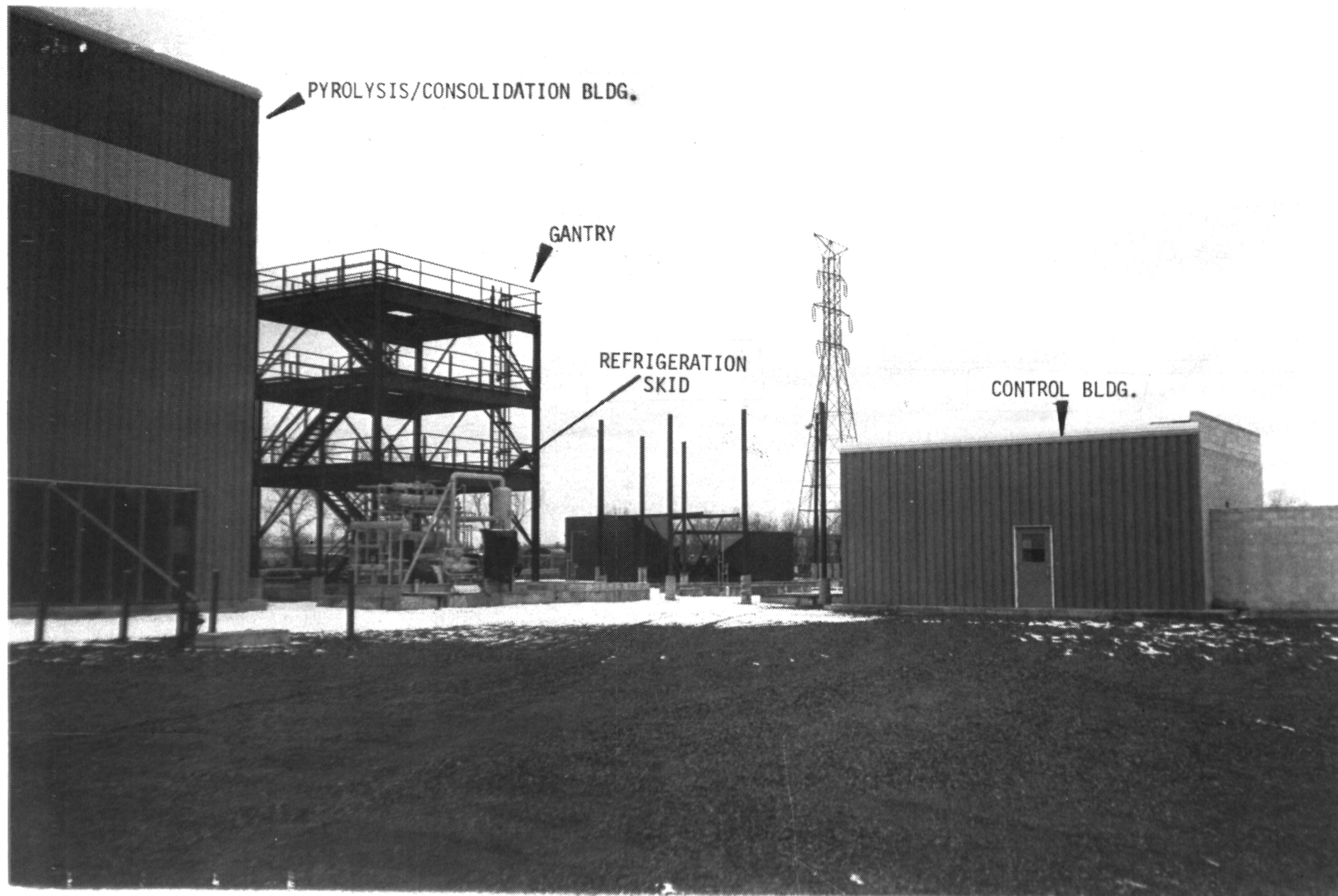
FIGURE 3.1 EPSDU Site
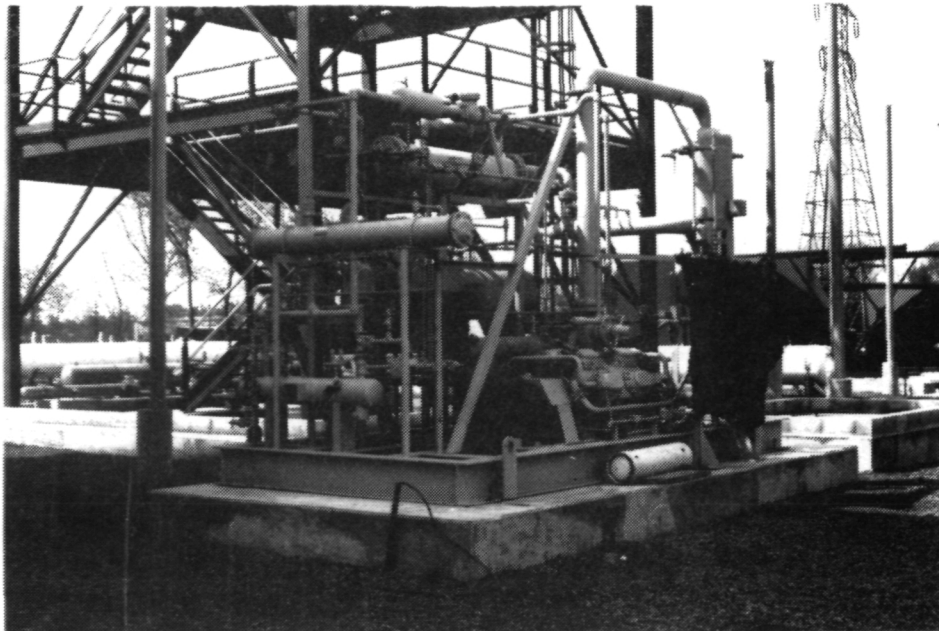
FIGURE 3.2

Refrigeration Skid
Equip. No. 469-12
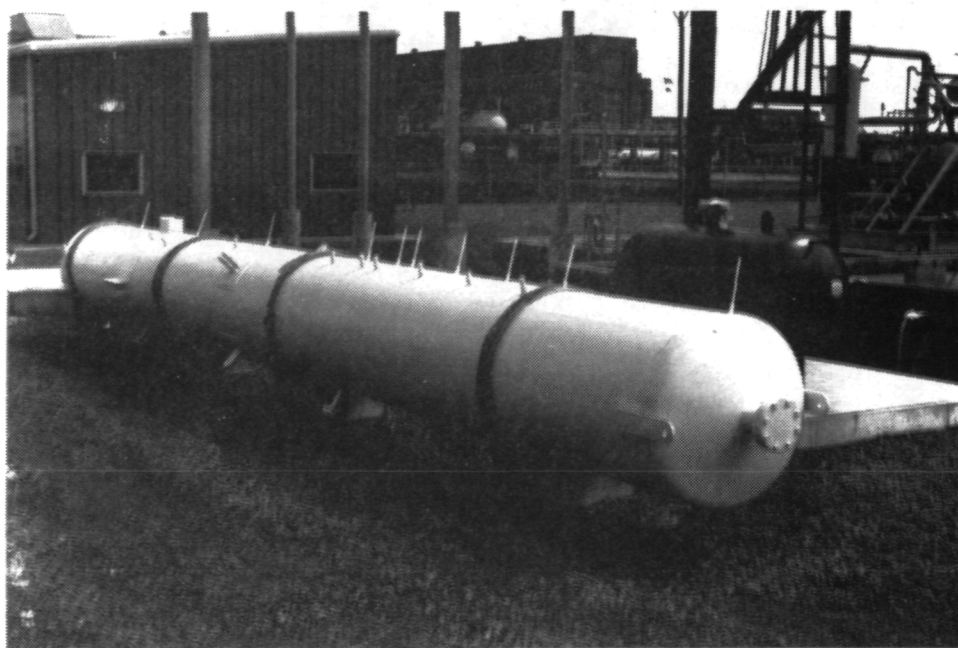

FIGURE 3.3

Dichlorosilane Colum
Equip. No. 432-06

FIGURE 3.4

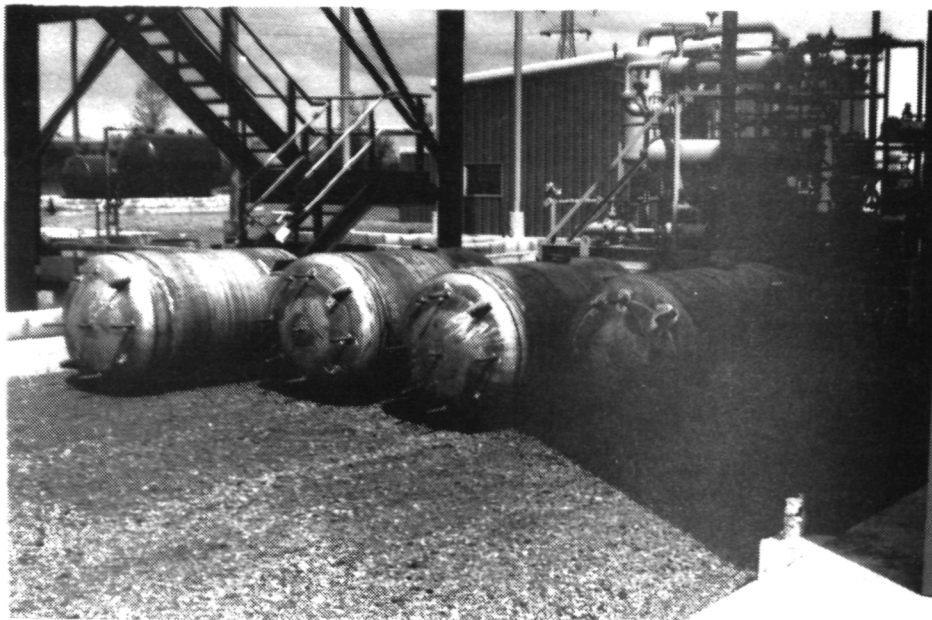Trichlorosilane Col
Equip. No. 432-04

FIGURE 3.5

Silane Storage Tanks

FIGURE 3.6

(1) Caustic Tank
(2) Silicon Tetrachloride
    Tank
(3) Trichlorosilane Tank

FIGURE 3.7

Super Heater
    and
Therminol Furnace

Subcontract bids were solicited from qualified companies to continue with the mechanical and electrical installation. The bids were within the expected allocations; however, none of these were awarded due to contract funding recisions.

1.4　OPERATION

　　The original intent of this work item was to cover all necessary activities to start-up and operate the EPSDU facility and demonstrate operability, produce representative product samples.　The performance data would be used to scale-up the equipment and system design to a commercial facility size and finally make an economic assessment for the production of silicon. Table I　shows the originally proposed report item broken down by task and work item.

　　The program delay has obviously delayed the operation phase of the program, however, activity had started in 1.4.1, Preparation.　This includes the writing of the Operating Manual and inspection of equipment received at the EPSDU site.

　　The Operating Manual has the following structure:

Volume I.　SAFETY

　　　1.1　Chemical Safety

　　　1.1.1　Chlorosilane and Silane Handling and Associated Hazards

　　　1.1.2　Chlorosilane Spill Control and Fire Prevention

　　　1.1.3　Handling of Acid and Solvent Cleaners

　　　1.1.4　Handling of Caustic Soda

　　　1.1.5　Handling of Waste Process Acids

　　　1.1.6　Handling of Free-space Silicon Powder

　　　1.1.7　Handling of Other Plant Chemicals

　　　1.1.8　Handling of Laboratory Chemicals

　　　1.2　Plant Safety

　　　1.2.1　Emergency Tactics & Firefighting

　　　1.2.2　Equipment Purging

　　　1.2.3　Protective Clothing

　　　1.2.4　Area Isolation & Plant Emergency Trips

Volume II:　DESCRIPTION

　　　2.1　Basic Process Chemistry

　　　2.1.1　Hydrogenation of Silicon Tetrachloride

Equipment Inspection

Major equipment items received at the EPSDU site were inspected by the EPSDU plant manager and by responsible process engineers. Although a specific inspection log was not maintained, a synopsis of the plant manager's notes and internal memorada was assembled and issued.

This serves as a deliverable "C" - equipment inspection and test report summary and is not included in this report. It should be noted that a detailed material receiving log has been maintained of equipment received at the site; this has been included with each monthly progress report. The log specifies purchase order number, a description, storage location, and tag number.

COMMERCIAL PROCESS ECONOMIC ANALYSIS

This report item was originally planned to develop process en-
gineering, design and cost estimate and subsequently prepare a detailed base-
line investment, operating cost and final product cost for a 100 MT/Year
commercial facility. This was based on the current EPSDU engineering design
information, and modifications based on supporting R&D information and EPSDU
operation were planned.

The work item was discontinued in 1980, at which stage the pro-
cess design had progressed to the point of issuing a detailed document titled,
"Process Functional Design Package for a 1000 MT/YR Commercial Polycrystal-
line Solar Silicon Plant". The package includes a process description,
process flow diagram, heat and mass balance, rough plant layout, process
functional specifications, and a list of design bases and assumptions.

The following sections present a brief summary of the process de-
sign package with a discussion of the features that are different from the EPSDU.
The expanded design package is included as Appendix B, however the Process
Equipment Functional Specifications (approximately 160 pages) have not been
included.

Commercial Process (Summary)

The process proposed for preparing semiconductor-grade silicon
from metallurgical-grade silicon is based on a well integrated arrangement of
purification steps that provide a cost-effective process system. The three
basic steps entail converting metallurgical-grade silicon to trichlorosilane,
redistribution of trichlorosilane to produce silane, and thermally decomposing
the silane to form polycrystalline silicon powder. The powder is then melted
and consolidated in a shotting apparatus to yield polycrystalline silicon
shot of a nominal 2 mm size.

Raw materials for the process consist of metallurgical-grade
silicon (98% pure) and copper catalyst. Hydrogen and silicon tetrachloride

must be added to replace the hydrogen and chlorine lost from recycle loops in the form of process wastes. The wastes to be disposed of include gaseous light impurities and heavy impurities composed of metal chlorides. Except for the raw material feed, waste disposal, and silicon-product streams, the entire process — hydrogenation, distillation, redistribution, and separation — is a closed-loop system in which purification is accomplished in each major step as process fluids are being recycled through the equipment. The process is shown in block-diagram form on Figure 5.1. The major steps are summarized in the following paragraphs.

M-G silicon and cement-copper catalyst are batch-fed to the hydrogeneration reactor which also receives the hydrogen and silicon tetrachloride recycle streams. The silicon is consumed to make trichlorosilane in an equilibrium reaction:

$$Si + 2H_2 + 3SiCl_4 \overset{Cu}{\underset{\rightharpoonup}{\leftharpoondown}} 4HSiCl_3$$
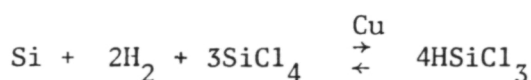
Metallic impurities in the silicon are converted to chlorides. Hot off-gases are quenched to their dew points to reject metal-chloride impurities, and then are condensed to ambient temperature. The non-condensible gases, predominantly hydrogen, are reheated and recycled to the reactor.

The condensed crude trichlorosilane, containing about 20% trichlorosilane, 80% silicon tetrachloride and some dissolved gases, is fed to the first of four distillation columns. In this column, gaseous light impurities are removed to the parts-per-billion level. In the second column, the bulk of the silicon tetrachloride is separated from other chlorosilanes and then is heated and recycled to the hydrogenation reactor. The other chlorosilanes from the second column are fed to the third column where trichlorosilane is separated from dichlorosilane.

The trichlorosilane bottoms product from the third column is redistributed to dichlorosilane and silicon tetrachloride in an equilibrium reaction using an amine-based resin catalyst:
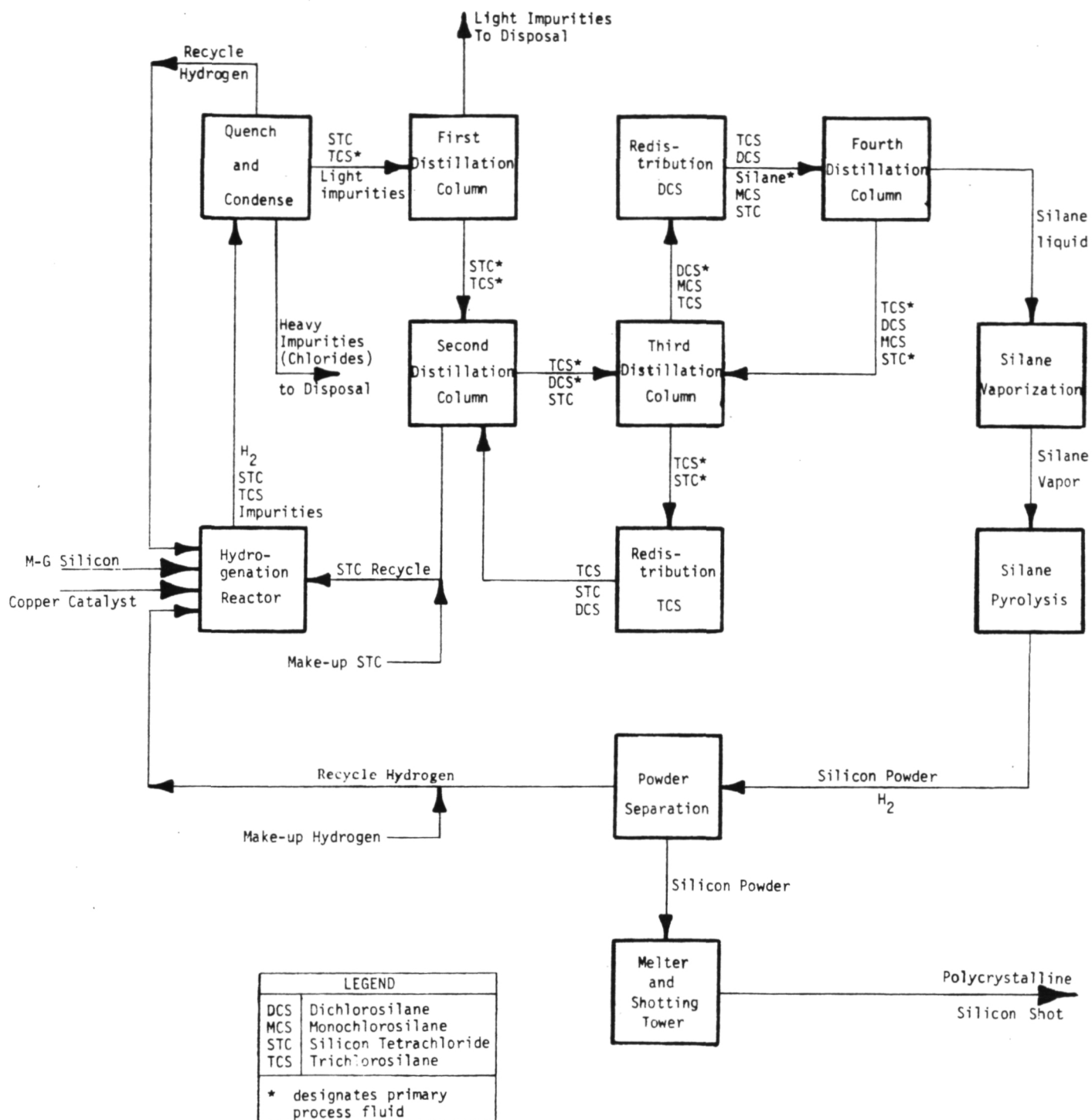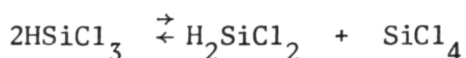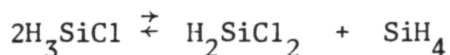
$$2HSiCl_3 \overset{\rightharpoonup}{\underset{\leftharpoondown}{}} H_2SiCl_2 + SiCl_4$$
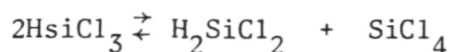
FIGURE 5.1 Silane-Silicon Process - Block Flow Diagram

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The redistribution products are recycled to the second column for separation.

The dichlorosilane distillate from the third column is redistributed to silane, monochlorosilane, trichlorosilane, and silicon tetrachloride in three reactions:

$$2H_3SiCl \rightleftharpoons H_2SiCl_2 \ + \ SiH_4$$

$$2H_2SiCl_2 \rightleftharpoons HSiCl_3 \ + \ H_3SiCl$$

$$2HSiCl_3 \rightleftharpoons H_2SiCl_2 \ + \ SiCl_4$$

The redistribution products are then fed to the fourth column for separation.

In the fourth column, silane is separated from the other process compounds which are returned to the third column. Gases lighter than dichlorosilane, including diborane, are removed in the first column, and gases heavier than silane, including diborane, are removed in the fourth column; any remaining traces of diborane which is closest in boiling point to silane, are removed to less than parts-per-billion levels in the fourth column. The silane is collected, tested for purity and vaporized to ambient temperature before being pyrolyzed.

In the pyrolysis reactor, the silane is expanded in a nozzle, heated, and irreversibly decomposed into silicon powder and hydrogen according to:
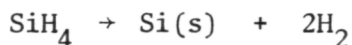
$$SiH_4 \rightarrow Si(s) \ + \ 2H_2$$

The hydrogen by-product is removed from the fine silicon powder and recycled to the hydrogenation section. The silicon powder is continuously fed into a heated quartz crucible where it is melted. The molten silicon is then dropped in a shotting apparatus to yield a nominal 2 mm spheroid and cooled to near ambient temperature before being removed as the final product of the process.

The gaseous and liquid process wastes are gathered in headers and processed for disposal. Wastes are thermally oxidized in a burner and then are scrubbed free of silica and hydrogen chloride. The waste by-products are scrubbed exhaust gas, silica powder, and muriatic acid.

Process Differences

There are four areas which are different from the EPSDU facility design. These are the quench condenser, the DCS distillation column, waste treatment and pyrolysis/consolidation. The differences and reasons for these differences are discussed below:

- Quench Condenser: The quench condenser is the heat exchanger in the hydrogenation section used to quench the hot off-gases. It will be used as a source of medium-level heat recovery. The liquid silicon tetrachloride, which is returned from the second distillation column to a storage tank, is used as the heat sink before being recycled into the hydrogenation reactor. Heat recovery will be accomplished by changing the quench condenser from a single counter-current heat exchanger to a double shell counter-current heat exchanger. (Functionally, two close-coupled heat exchangers with the tube bundles in series.) The first shell will cool the hydrogenation gases (STC, TCS and H_2) from the hydrogenation reactor against the silicon tetrachloride (STC) feed to the vaporizer. The STC feed will exit the first shell as a pre-heated liquid at 340°F and be fed into the vaporizer. The hydrogenation gases will be quenched to 320°F in the first shell of the exchanger. This stream will then be quenched to 100°F in the second shell using cooling water as the heat sink. The heat recovery will be about 1.9 million BTU/Hr. The heat recovered goes directly into preheating the STC before the vaporizer. This will reduce the vaporizer duty since a significant portion of its duty is used to pre-heat the STC.

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- Dichlorosilane Column: The functional design of the third distillation column (DCS column) which separates trichlorosilane has also been altered from the design used in EPSDU. The DCS column will operate at 175 psia rather than the 320 psia operating pressure specified for EPSDU. This will result in an easier separation (fewer trays), a lower design temperature, and consequently, a lower capital cost.

- Waste Treatment: The waste treatment area of the commercial facility will produce 20% muriatic acid as a by-product. The design of this area will revert to the original design of the EPSDU waste treatment. The acidic waste water from EPSDU will be neutralized before being released to the sewer. This approach was used for EPSDU because it was economically impractical to produce muriatic acid. However, the commercial economics indicate that the production of muriatic acid as a by-product, is a cost-effective means of waste disposal, and it also reduces the environmental load of the plant.

- Pyrolysis Consolidation: The conceptual design of the pyrolysis/consolidation area will remain essentially the same as EPSDU. However, an effort was made in the commercial functional package to specify all components of this section of the facility, including those not yet specified in the EPSDU design. This was done so that the cost estimate will reasonably reflect a silicon-shot product instead of a molten product. The functional specifications are based on in-hand knowledge and various assumptions about the success of the free-space reactor and melter R&D programs. There will be six pyrolysis reactors which will perform functionally as previously described. The silicon powder from all six of the reactor hoppers is transported pneumatically into a central bin. This bin feeds twelve melter assemblies by dense-phase

pneumatic transport. The melter assemblies consist of a powder receiving bin, a powder screw feeder, a crucible/furnace melting unit and a shotting tower. The receiving bin, screw feeder, and crucible/furnace unit, receive the powder, melt it, and form silicon shot through an orifice in the bottom of the crucible. The shot cools as it falls through the shotting tower and is caught in a product hopper in the bottom of the tower. The shot from all of the melters is then transported, using a drag conveyor, to a central bin from which the silicon-shot product is packed into drums.

The major process equipment not included in the four areas noted above will remain functionally the same as the equipment designed for EPSDU.

1.6　　　PROCESS SUPPORT R&D

The supporting R&D program is separate from the mainstream design
effort and includes all activities associated with:

- Analytical and experimental development of the free-space
  reactor (FSR).

- Design and development of a melting/consolidation system.

- Fluid-bed reactor (FBR) development.

- Quality control techniques and procedures.

Information generated in this program was used for the EPSDU program and the
commercial facility economic analysis.

1.6.1　　　FREE—SPACE REACTOR DEVELOPMENT

Program Objective

Construction and operation of the Tonawanda free-space reactor
PDU had two explicit objectives: to demonstrate that a free-space reactor
can operate reliably for extended periods, and to investigate the silicon
product powder purity. Achieving these objectives would confirm the via-
bility of the free-space reactor concept for EPSDU while providing a rational
basis for scale-up of the actual EPSDU reactor.

Introduction

Free-space silane pyrolysis technology has been advanced signi-
ficantly as a result of the JPL-sponsored development effort at Union Car-
bide. This activity builds on earlier UCC-funded work involving the construc-
tion, operation, and testing of a Free-Space Reactor Process Development
Unit (FSR PDU). The testing at the Parma Technical Center demonstrated the
feasibility of producing semiconductor-grade polycrystalline silicon by
pyrolyzing high purity silane.

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A similar reactor with improved modifications was developed and tested at UCC Tonawanda facility. A series of 19 test runs were made which confirmed that the free-space reactor is viable and an acceptable grade of silicon powder can be obtained.

Conclusions

The experimental work resulted in the following conclusions which influenced the design of the EPSDU Free-Space Reactor.

It was demonstrated that the occurence of hard silicon deposits on the reactor wall (observed during early runs) could be eliminated by maintaining the peak reaction zone temperature (12 inches below the head) between 830° and $900^o C$. This was achieved by utilizing an induction heater coil with variable pitch windings (close pitch at each end and wider pitch at the center). The conversion of silane was in excess of 99.99%. Average powder particle size and bulk density range were found to be 0.34 μm and 2.3 to 8.6 lb/ft^3 respectively.

New improved mechanical features - scraper seal, quartz liner support system and powder transport system - demonstrated operational feasibility and durability. All of these features were used in the design of the EPSDU FSR.

Powder purity was not conclusively demonstrated due to the inadequacy of the sample preparation and analytical techniques used (Atomic Adsorption and Inductively Coupled Plasma Emission Spectroscopy). However, a polysilicon boule pulled from melted FSR powder was shown to have 55 Ω cm P-type resistivity.

In summary, the test program was very successful and provided a firm basis and understanding to confidently scale-up the EPSDU design.

The development work carried out on free-space pyrolysis showed it to be a viable technique for decomposing silane. It can be carried out in

a compact, high-throughput reactor capable of producing high-purity silicon powder. However, this powder requires a further processing step of consolidation to enable handling with minimum contamination.

1.6.1.1   PDU Experiments

<u>Free-Space Reactor Design and Assembly</u>

A free-space reactor PDU was designed and installed in an explosion-proof test facility in our Tonawanda laboratories. Initial improvement over the previous PDU at Parma included:

- <u>Free-Space Reactor Shell</u> - The shell, shipped from Parma, was made from Monel and was substantially de-alloyed, especially in a 1-foot section of the hot zone. Flakes of zinc had accumulated on the outside of the reactor cylinder and the wall was less than half of the original thickness. A new reactor shell was fabricated from Incoloy 800 since this alloy is metallurgically much better suited for this application.

- <u>Silicon Hopper</u> - A large hopper was designed and fabricated to hold a 2½-day inventory of silicon powder.

- <u>Reactor Head Flange</u> - The top head assembly for the pyrolysis reactor was redesigned to incorporate a remote controlled actuator for a mechanical scraper. A small feeder cone was also designed for the bottom of the silicon product hopper to faciliate withdrawing powder by adding fluidization gas.

- <u>Induction Power Supply</u> - New capacitors were installed in the induction heating power supply to replace the previous PCB-filled units which are environmentally unacceptable.

Figures 6.1 and 6.2 show the silicon hopper design and the flow diagram for silane pyrolysis, respectively. The process and instrumentation diagram (P&ID) is shown as Figure 6.3 and identifies all required instrumentation for control and operation of the free-space reactor.
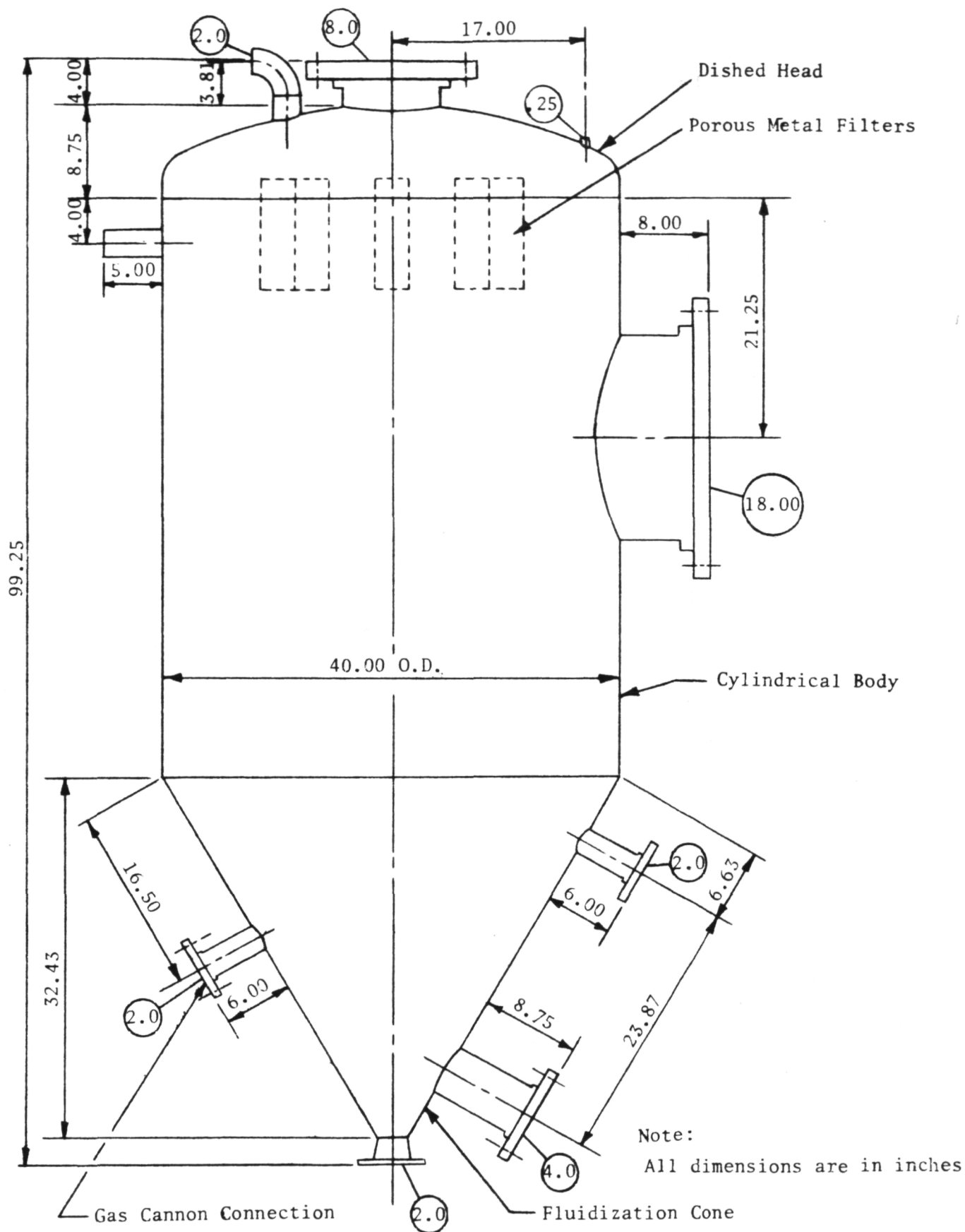
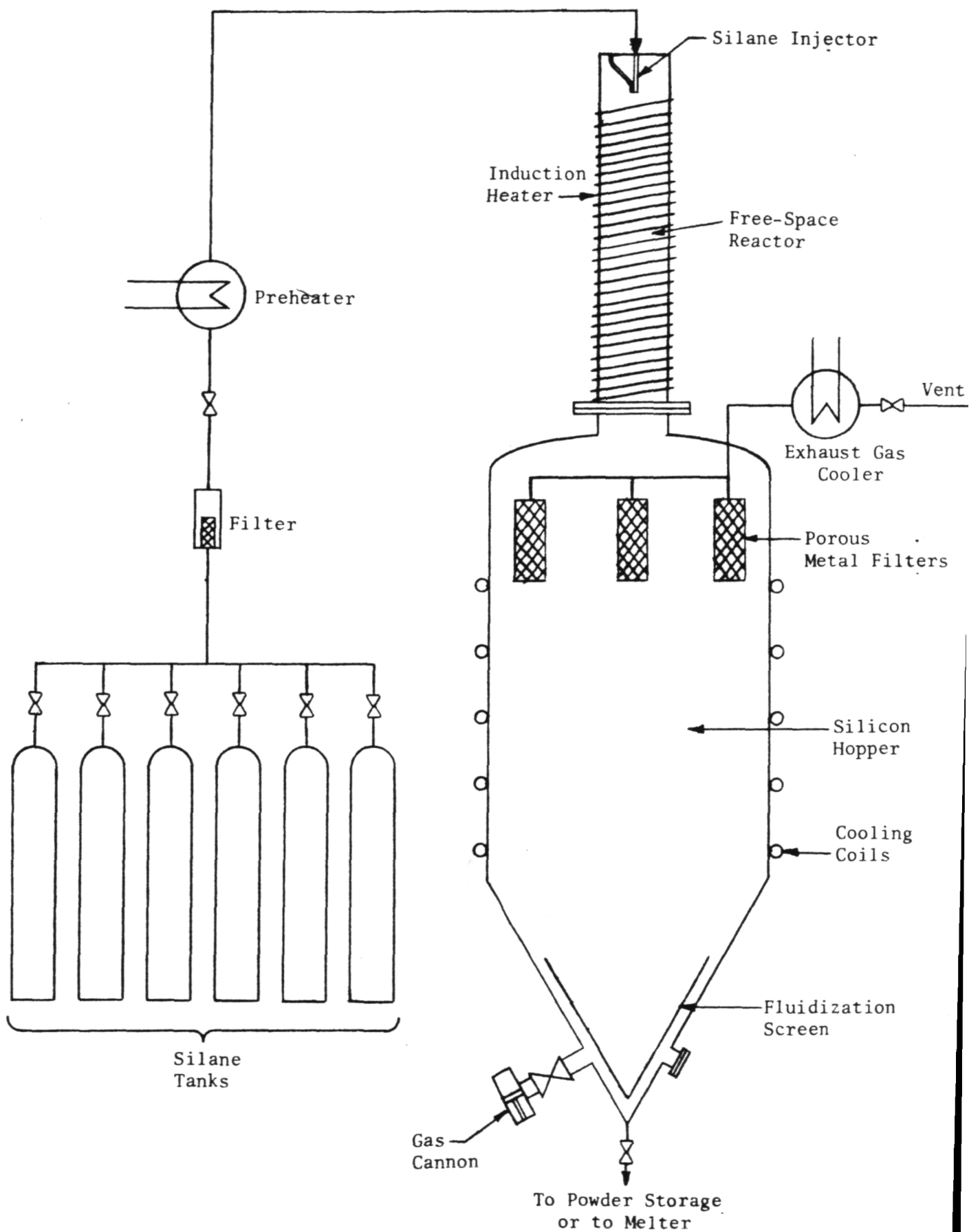FIGURE 6.1 Silicon Hopper for Free-Space Reactor, PDU

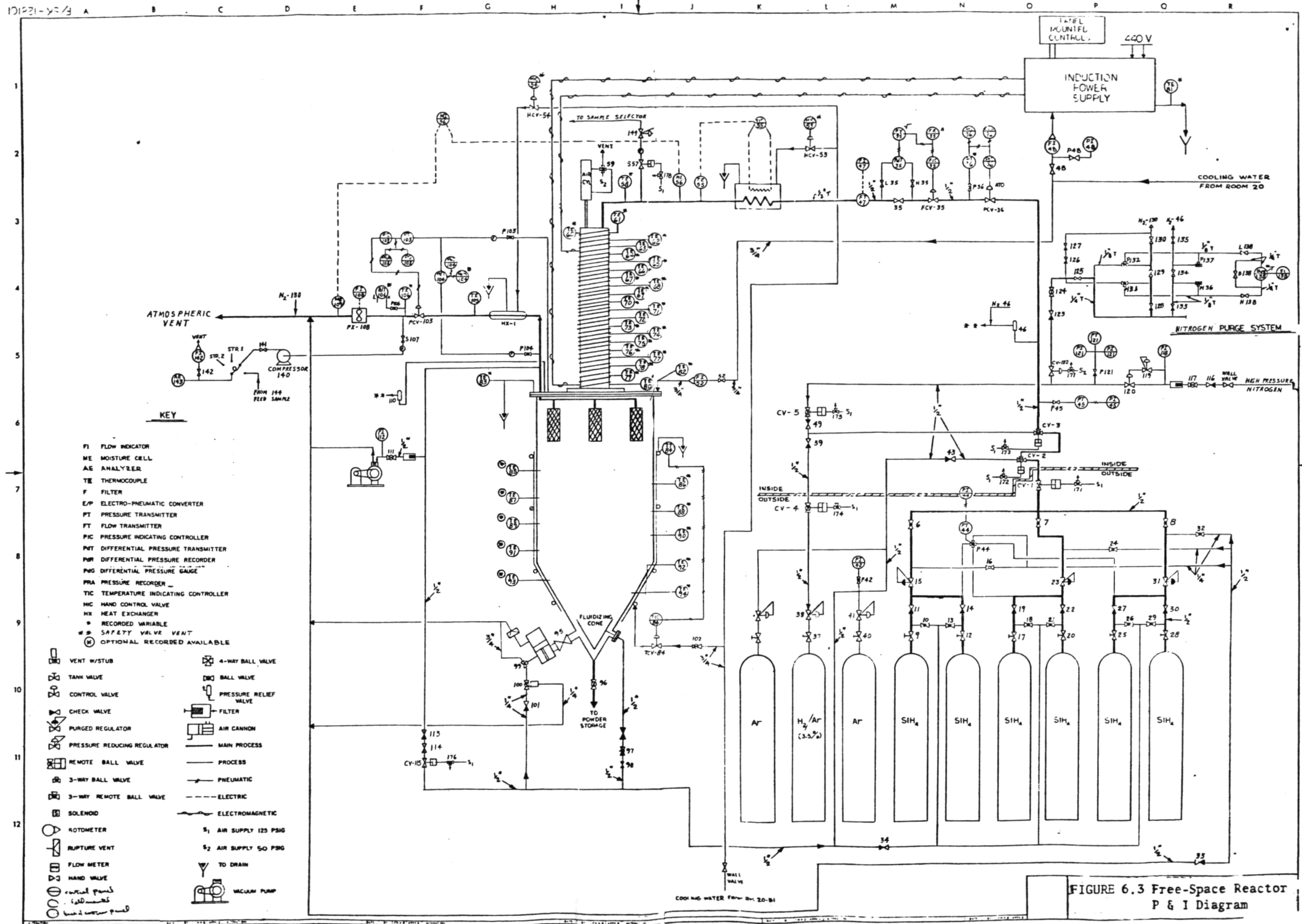Figure 6.2 Flow Diagram for Silane Pyrolysis Experiments in Free-Space Reactor

FIGURE 6.3 Free-Space Reactor
P & I Diagram

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The mechanical assembly of the PDU was completed in the fourth quarter of 1979 and system checkout was begun in the first quarter of 1980. Figure 6.4 through 6.7 show the free-space reactor, silicon powder hopper, and other pieces of equipment which comprise the Process Design Unit (PDU). Figure 6.8 and 6.9 show the induction heater motor generator and the control panel, respectively.

Initial Testing, Experimental Runs (1 through 7)

The seven runs made during this program are summarized in Table VI.

The properties of the powder produced in the PDU were as follows: Powder bulk density proved to be sensitive to both reactor operating temperature and throughput. The lowest density recorded was for operation at 0.56 kg/hr and 900°C (0.076 g/cm^3) and the highest was 1.95 kg/hr at 870°C (0.27 g/cm^3). Operation at higher throughputs and temperatures showed temperature to be the dominant factor as bulk density decreased. Evaluation based on scanning electron microscope micrographs indicated average particle diameter between 0.3 and 0.4 μm. A single B.E.T. surface-area determination yielded a calculated diameter of 0.9 μm.

Powder purity was a problem during this early work. Levels of metallic impurities generally were in the low ppm range. The probable source of these impurities was the reactor wall which was exposed to the powder when the quartz liner broke and later when the liner was removed to avoid the operating problems causing by liner breakage. Direct attack on the wall was observed in the reactor hot zone with Incoloy peeling off the wall in strips. Alloying with silicon and chloride corrosion (from chlorosilanes in the silane) were the probable causes. The major cause of liner breakage was the formation of dense, mechanically-strong silicon deposits in the upper section of the reactor. These resisted passage of the scraper and either caused the liner to fail in tension from induced hoop stress or created sufficient drag for the scraper to lift the liner off of its supports and slam it against the head of the reactor. Computer modeling of the reactor indicated that the deposits re-

Silane Inlet – SS Flex Hose

Scraper Assembly

Air Cylinder

Silane Injector

Cooling Water Lines
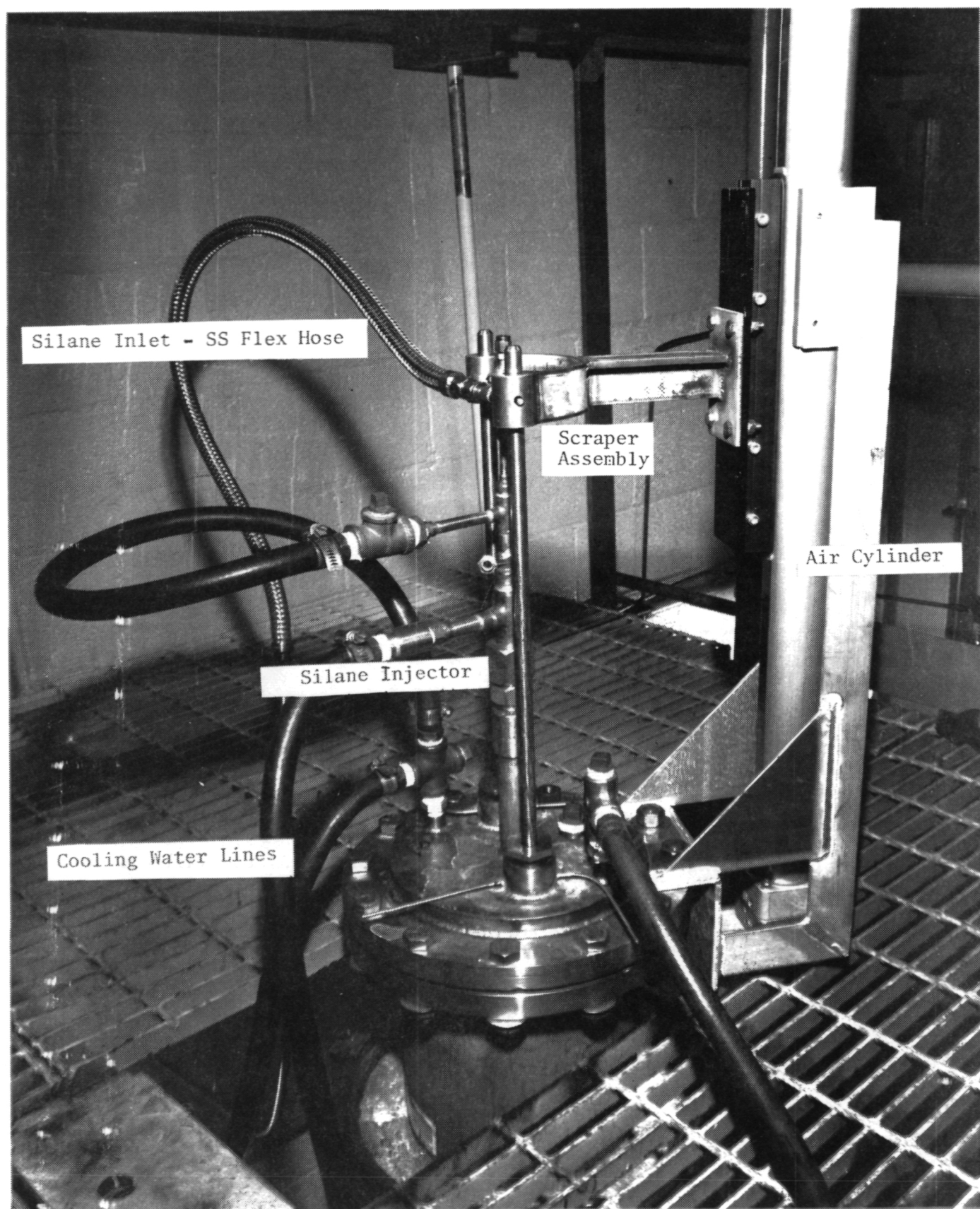
FIGURE 6.4   Free-Space Reactor, Top Flange Assembly

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FIGURE 6.5 Free-Space Reactor and Silicon Powder Hopper

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Spare Port

Lower Section of Silicon Powder Hopper

Port to Fluidizing Cone

Powder Discharge Valve

FIGURE 6.6 Bottom Half of the Silicon Powder Hopper

Gas 'Cannon' on Silicon Hopper

FIGURE 6.7   Gas Cannon on the Silicon Powder Hopper

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FIGURE 6.8   Induction Heater Motor-Generator for Free-Space PDU
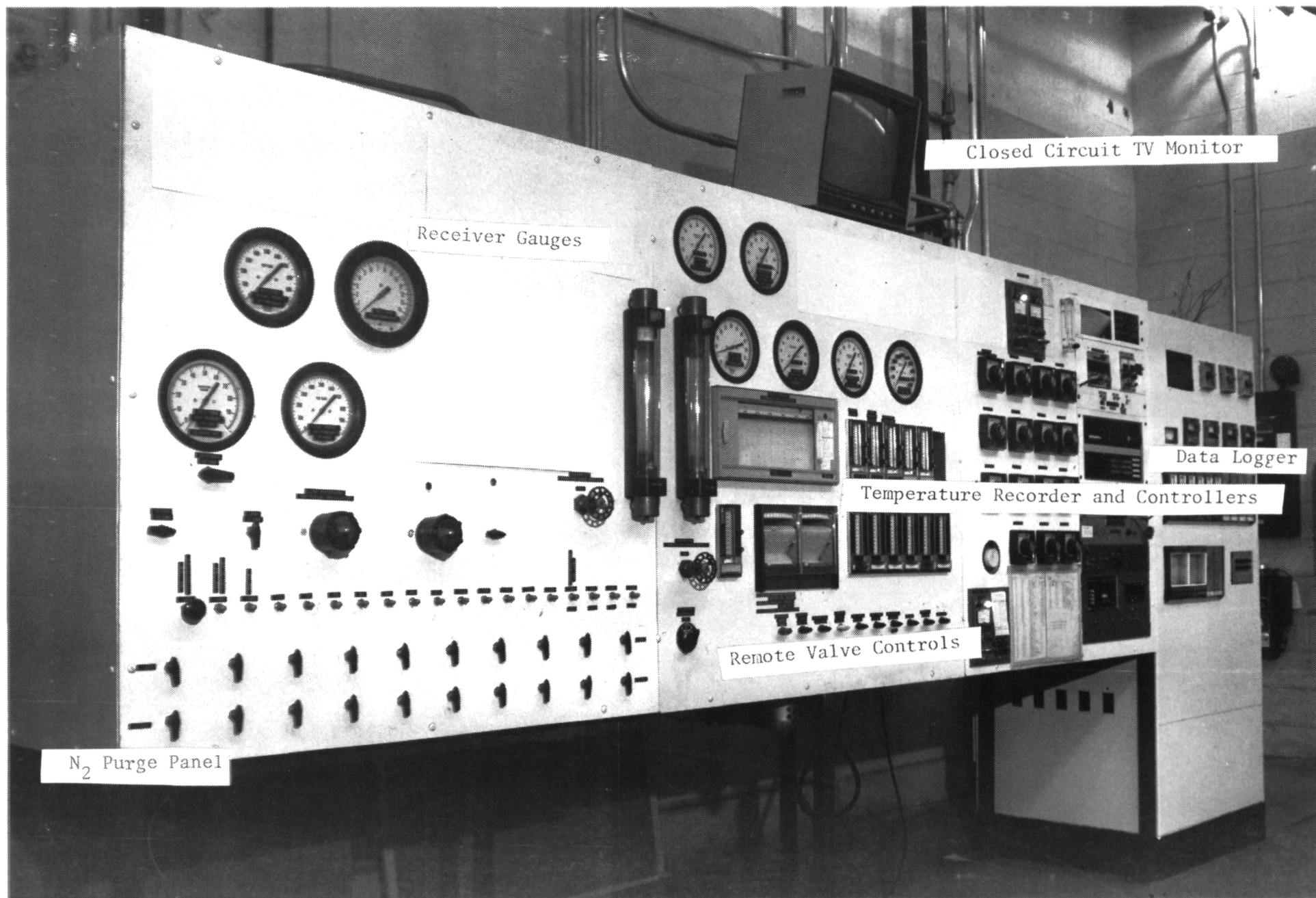
FIGURE 6.9 Control Panel for the Free-Space PDU

TABLE VI

FSR PDU RUN SUMMARIES (1 through 7)

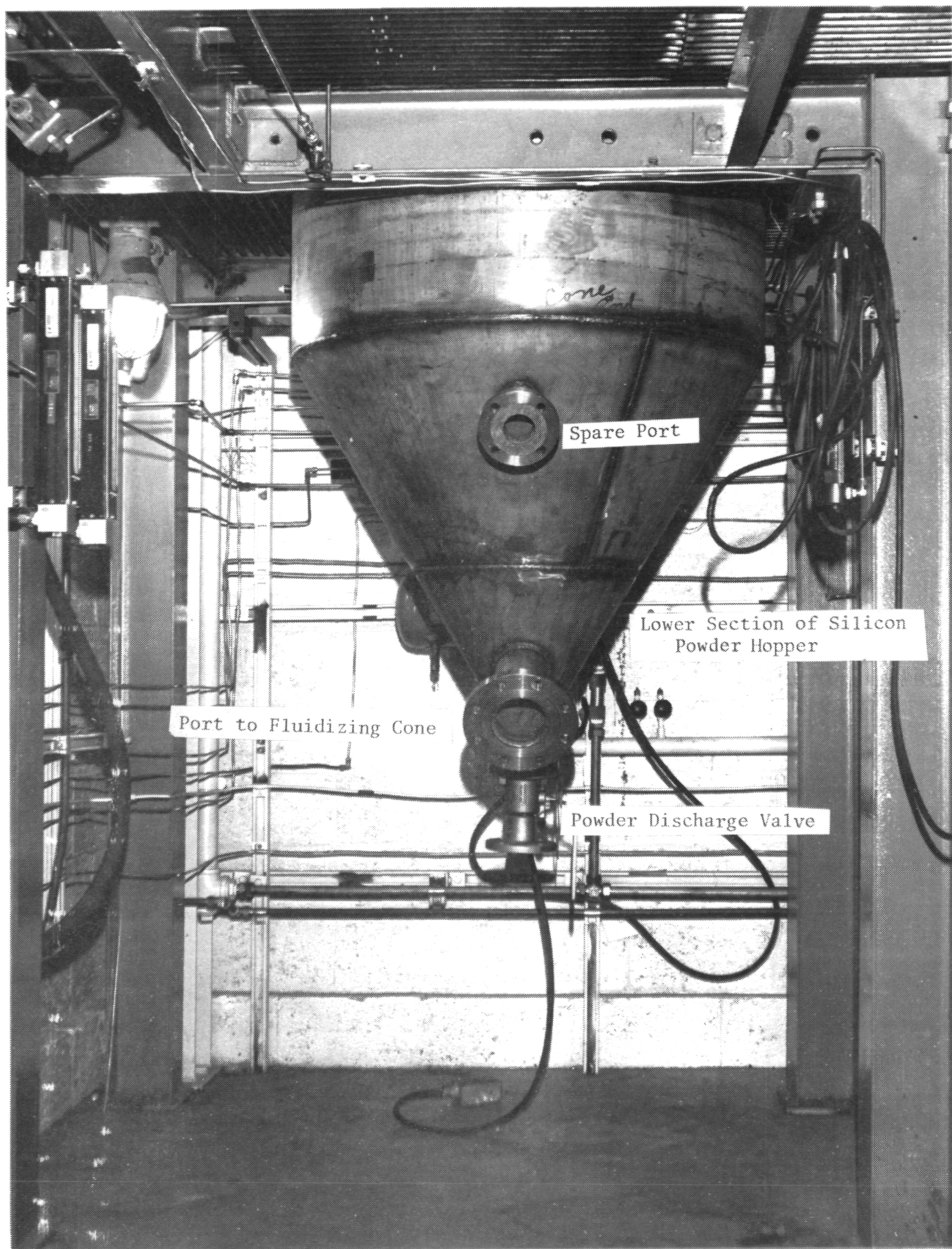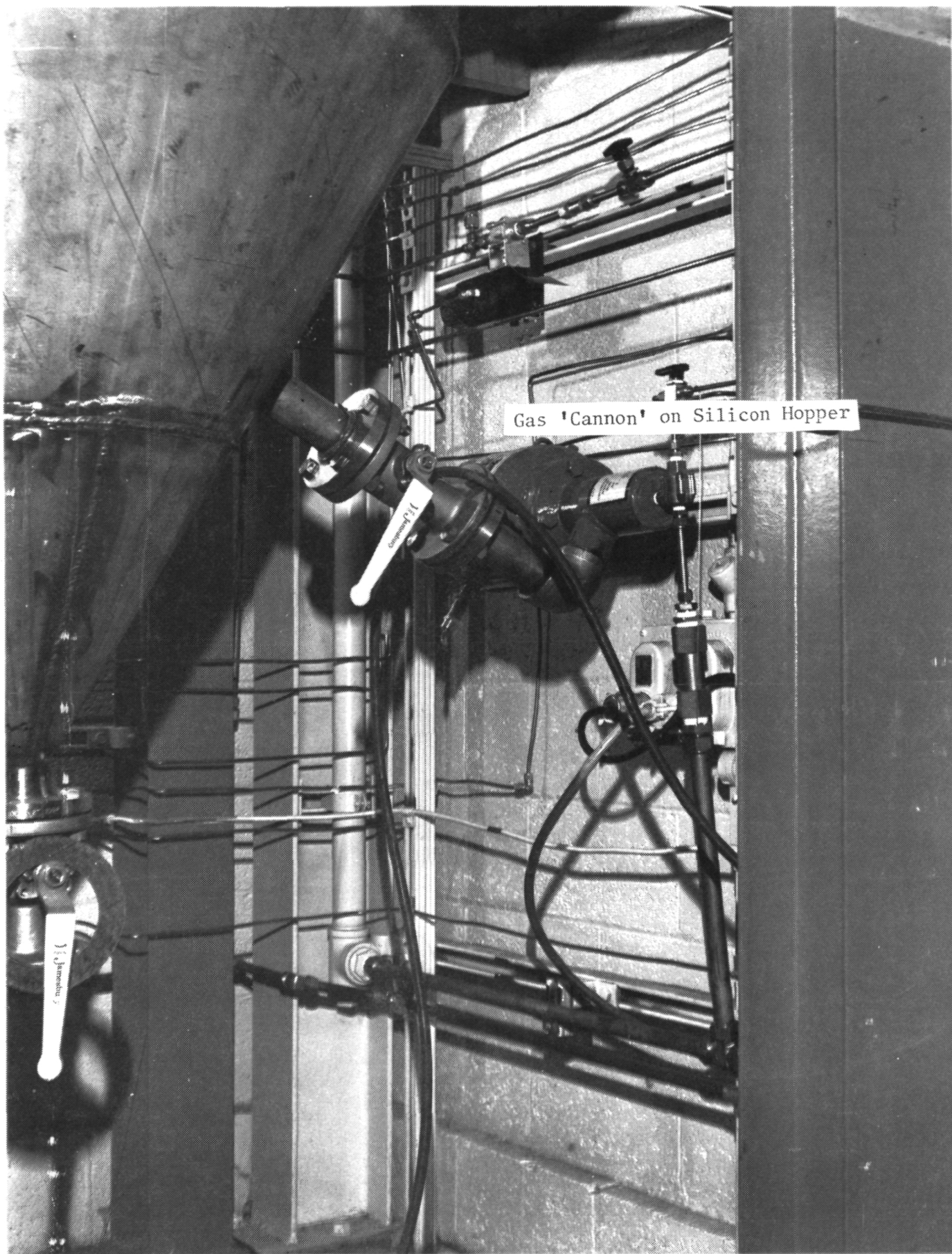| Run No.	Date	Temp.	Press.	Flow Rate	Duration	Powder Bulk Density	Fe	Ni	Cr	Mn	Comments
1	1/13/80	900°C (1650°F)	142.7 KPa (20.7 psia)	0.56 kg/hr (1.24 lbm/hr)	1.0 hr	0.075 gm/cm³ (4.72 lbm/ft³)	130	21	--	3	Shakedown run. No scraper. Reactor not cleaned. Regulator leaked.
2	3/4/80	850°C (1560°F)	142.7 KPa (20.7 psia)	2.25 kg/hr (4.95 lbm/hr)	7.5 hrs	0.25 gm/cm³ (16.1 lbm/ft³)					Scraper jammed at 7.5 hrs liner broken - powder exposed to atmosphere
3	3/21/80	870°C (1600°F)	142.7 KPa (20.7 psia)	1.95 kg/hr (4.30 lbm/hr)	24 hrs	0.27 gm/cm³ (16.8 lbm/ft³)	4	2	2	0.5	Major milestone. System precleaned - DI H₂O Liner Broken. Some powder exposed to atmosphere
4	4/16/80	1100°C (2010°F)	142.7 KPa (20.7 psia)	4.5 kg/hr (10.0 lbm/hr)	2.5 hrs	0.22 gm/cm³ (13.5 lbm/ft³)	8	2	2	4	Throughput test - No liner. Thermocouple failure led to operation at higher than planned temperature. System not opened at end of run.
5	4/18/80	960°C (1760°F)	142.7 KPa (20.7 psia)	6.2 kg/hr (13.7 lbm/hr)	1.5 hrs	0.167 gm/cm³ (10.5 lbm/ft³)	8	3	3	4	Throughput test - No liner. No problems. Conversion fell off at 13.7 lbm/hr. System not opened.
6	4/25/80	950,885°C (1750,1625°F)	142.7 KPa (20.7 psia)	4.5 kg/hr (10.10 lbm/hr)	2.5 hrs	0.154 g/cm³ (9.6 lbm/ft³)	2	2	1	5	Throughput test. No liner. System not opened.
7	4/20/80	930°C (1710°F)	142.7 KPa (20.7 psia)	2.7 kg/hr (6.0 lbm/hr)	24.5 hrs	0.208 (13.0 lbm/ft³)	13	2	1	3	24 hour run. Very smooth. Minimal temperature control problems. No Liner. Major Milestone. System opened for inspection.

Purity (PPM)

sulted from the sharply non-uniform wall-temperature profile of the current reactor which allowed significant quantities of silane to penetrate the recirculating flow zone close to the wall. The deposits formed where temperature and concentration conditions at the wall favored heterogeneous silicon deposition over homogeneous reaction.

Other impurities were introduced as a direct result of the deposits. After the deposits were dislodged from the reactor wall by the scraper, they blocked the outlet from the hopper. Consequently the powder had to be removed through the manway which permitted heavy exposure to atmosphere. The 24-hour runs demonstrated that the PDU could operate successfully for extended periods at a high silane throughput and a high conversion efficiency without plugging or otherwise becoming inoperable. In addition, the short-term capacity tests were successful in determining the maximum throughput capability of the PDU as currently configured. Figure 6.10 summarizes the conversion efficiencies obtained for all seven runs and plots these efficiencies against the reactor throughput. On the basis of a 98% conversion efficiency criterion, the PDU is capable of processing about 4.5 kg/hr (10.0 lb/hr) of silane.

Experimental Runs (8 through 13)

Six runs were made during the second period as summarized in Table VII. Installation of a new insulation blanket and repositioning of the induction coil increased the reactor wall temperature to $400^{\circ}C$ in the upper reactor and greatly improved reactor performance. No silane was detected on the chromatograph during any run even with maximum wall temperatures as low as $870^{\circ}C$ (Run 8).

Before Runs 11, 12, and 13 were made, a new induction heating coil, which uses non-uniform coil spacing to concentrate energy in the crucial upper section of the reactor, was installed and wall temperature profiles much superior to those obtained with the previous coil were produced as shown in Figure 6.11. The silane injection nozzle was also moved 3.8 cm (1.5 in.) further down the reactor to enable the silane jet to enter into a higher temperature region. Run 11 was made at two flow rates: 1 hour at 4.3 kg/hr
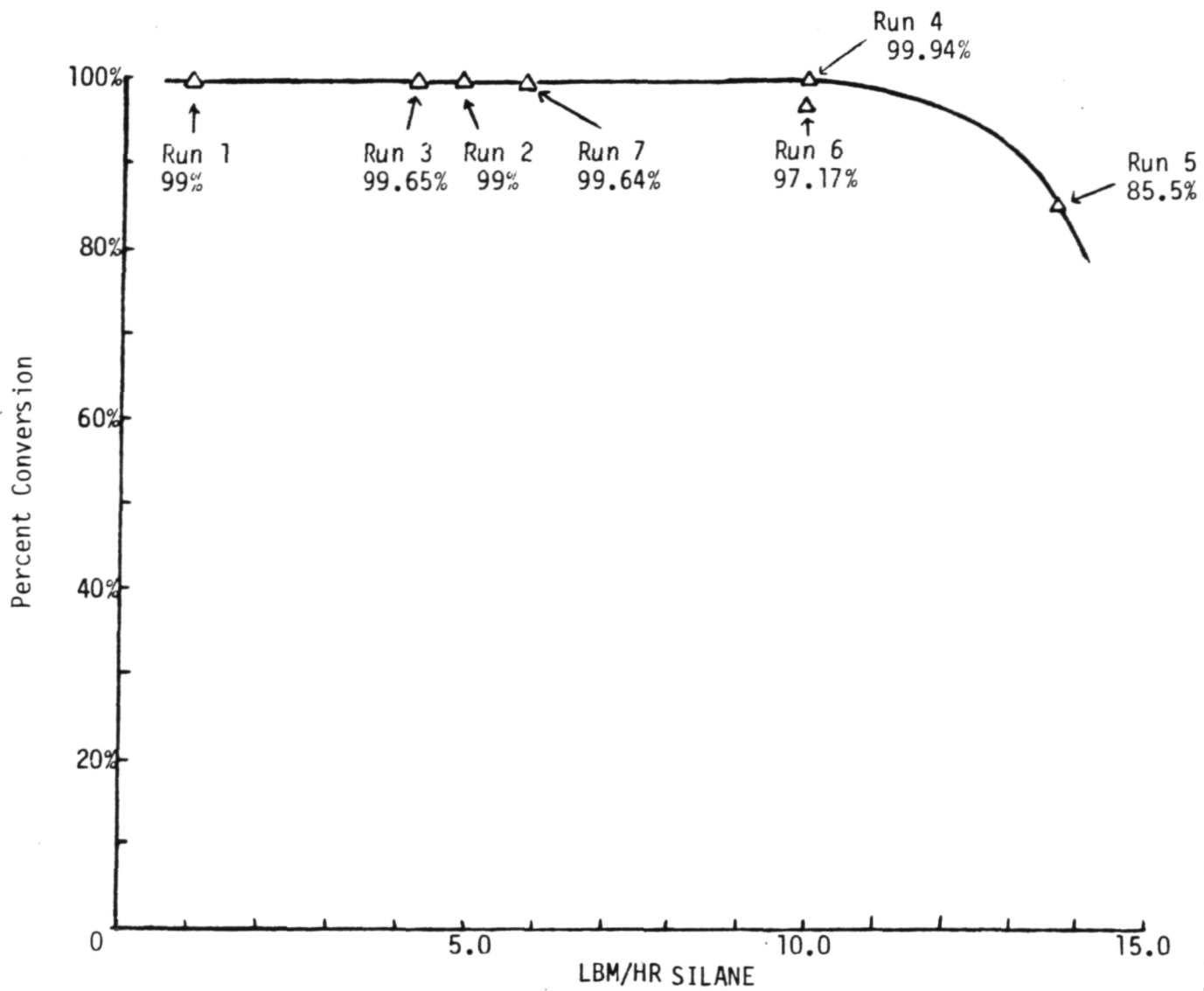
FIGURE 6.10   FSR PDU Throughput Capacity

TABLE VII

FSR RUN SUMMARIES

RUN NO.	DATE	DURATION (hr)	SILANE FLOW (kg/hr)	MAX WALL TEMPERATURE Deg.C	PRESSURE (Kpa)	DEPOSITS	BULK DENSITY (gm/Cm^3)
8	7/ 9/80	2.0 hrs	2.3 kg/hr	870°C	138 Kpa	No	0.137 g/cm^3
9	7/22/80	2.0 hrs	2.3 kg/hr	940°C	138 Kpa	No	0.077 g/cm^3
10	7/25/80	2.25 hrs	2.4 kg/hr	915°C	276 Kpa	No	0.113 g/cm^3
11	8/14/80	2.0 hrs	3.1 kg/hr (6.8 lb/hr)	905°C[1]	138 Kpa	No	0.085 gm/cm^3
12	8/18/80	3.1 hrs	2.8 kg/hr (6.1 lb/hr)	960°C	138 Kpa	No	0.078 gm/cm^3
13	8/26/80	12.0 hrs	2.2 kg/hr (4.8 lb/hr)	950°C	138 Kpa	No	0.036 gm/cm^{3}[2]

NOTES: (1) Run terminated when lower reactor wall temperature exceeded 1030°C .

(2) Skimmed from top of powder bed in hopper. Average density was approximately 50% higher, or 0.054 gm/cm^3 (3.4 lb/ft^3). Average wall temperatures higher in run 13 than in run 12, although the maximum wall temperature was slightly lower.

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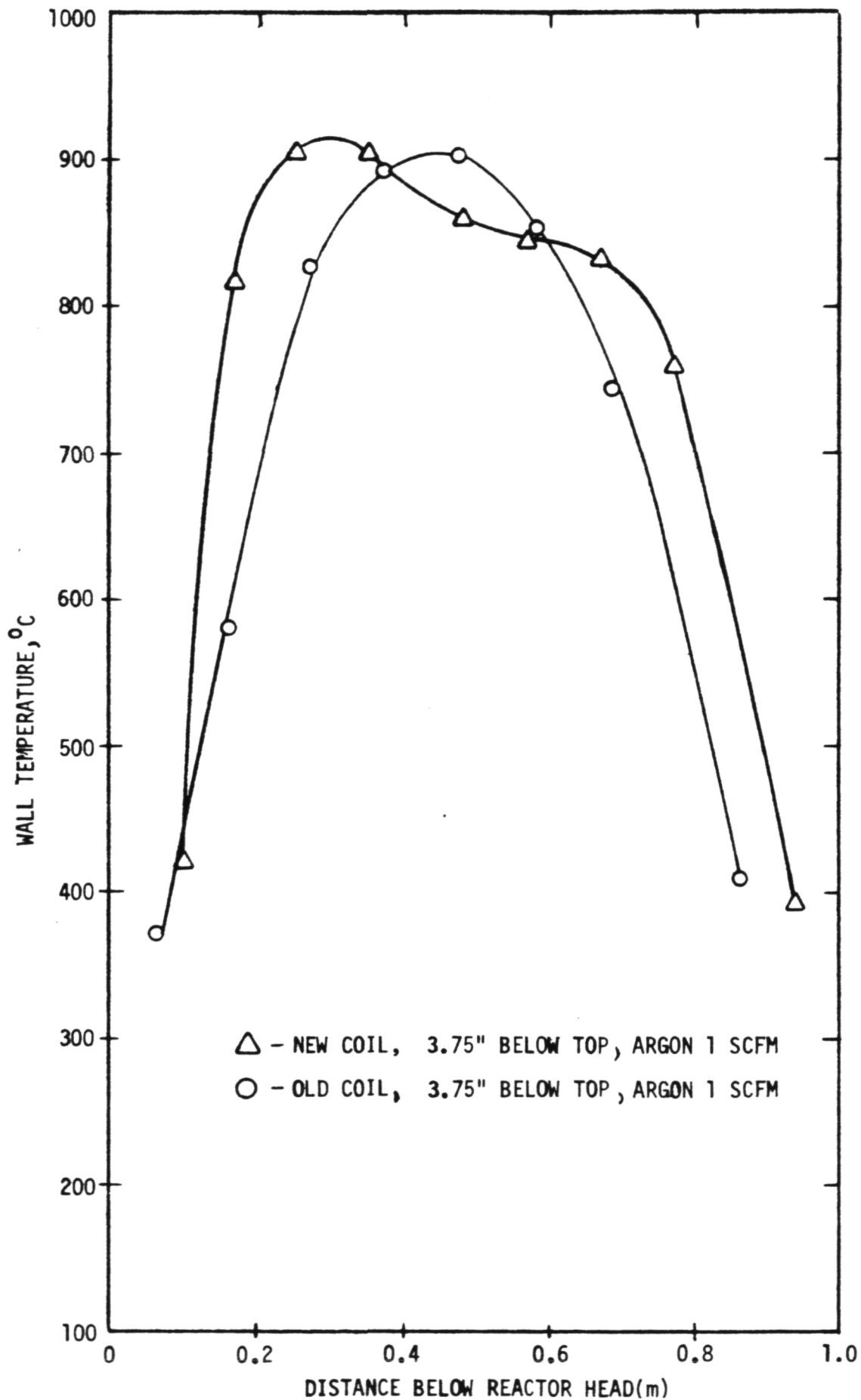
FIGURE 6.11   FSR Temperature Profile
Old Induction Coil Vs. New Coil

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(9.5 lb/hr) and 43 minutes at 2.5 kg/hr (5.6 lb/hr). The run was terminated when the wall temperature in the lower half of the reactor exceeded 1030°C. Powder buildup on the wall below the scraper, combined with a high power input from the new coil, created this rise. Inspection of the reactor with a bore-scope showed no buildup of powder on the wall or injection nozzle.

To reduce the wall temperature, the insulating blanket was removed from the bottom 7 inches of the reactor. Run 12 was successful at flow rates of 2.3 kg/hr (5.2 lb/hr) for 2.4 hours and 4.1 kg/hr (9.1 lb/hr) for 0.7 hour. Very little powder buildup was noticed after run completion.

Run 13 was a 12-hour test using new, purer silane. No operational difficulties were encountered and this was the smoothest run made in the free-space reactor. One drum (19.2 lb) of powder was shipped to Kayex Corporation in Rochester, New York, for melting, doping, and crystal pulling. Inspection of the system showed no hard deposits on the reactor wall or injection nozzle, which indicates very clearly that hard wall deposits have been eliminated in the PDU.

The successful completion of five short duration and one long-duration runs without any evidence of hard wall-deposit formation is an extremely siginificant accomplishment. The design of the EPSDU reactor can now proceed with confidence that the free-space reactor concept is workable.

Purity of powder from Run 11 and a sample of high quality polysilicon obtained from Dow-Corning were compared using the atomic absorption spectroscopy method. The AA method differed from previous analyses in that ultra-high purity HF and high purity HNO_3 were used to dissolve the silicon. The level of impurities found in the powder and polysilicon thus appeared to depend strongly upon the analytical method used. To confirm this supposition, samples of powder from Run 13 and a sample of Dow-Corning polysilicon were sent to Union Carbide's Tarrytown laboratory for analysis on an Induction-Coupled Plasma (ICP) spectrophotometer. The results of this analysis and of a conventional AA analysis made at Tonawanda are summarized in Table VIII and are comparable to those of earlier runs. The variation from sample to sample

and the high levels of impurities found in what should have been an exceedingly pure polysilicon chunk imply that handling or impure reagents may have been responsible for most of the observed impurities. X-ray diffraction analysis of powder from Run 13 showed that the powder has a crystalline structure.

TABLE VIII

ANALYSIS OF RUN 13 POWDER

Method	Sample	Fe	Impurities (PPM) Ni	Cr	Mn	Zn
AA*	1	1.0	0.4	0.2	0.1	-
	2	13.0	0.7	0.4	0.5	-
	Polysilicon "Chunk"[1]	0.75	0.15	0.1	0.1	0.15
ICP	3	0.6	<0.5	<0.5	0.2	<0.3
	4	3.0	<0.5	<0.5	0.2	<0.3
	Polysilicon "Chunk"[1]	1.0	<0.5	<0.5	<0.1	0.6

* Cr, Ni, Mn by graphite furnace AA; Fe by flame AA.

[1] Dow-Corning supplied. These levels are obviously high. Chunk prepared by surface etching.

Following Run 13, a new powder-transport system was designed, fabricated, and installed in the hopper. The system is similar to the approach used in the blow-pot based pneumatic conveying systems. A truncated porous media sits in the bottom of the hopper and powder is removed through a vertical 1.5 inch pipe that exits through an unused port.

A new reactor liner support (Figure 6.12) was also installed which utilizes Inconel springs and a self-centering mechanism to hold the quartz liner firmly against the reactor head and compensates for thermal expansion during operation. An inert gas purge in the annular space between the liner and reactor further guards against powder infiltration. This support should eliminate the observed problem of attack by the powder on the Incoloy wall.
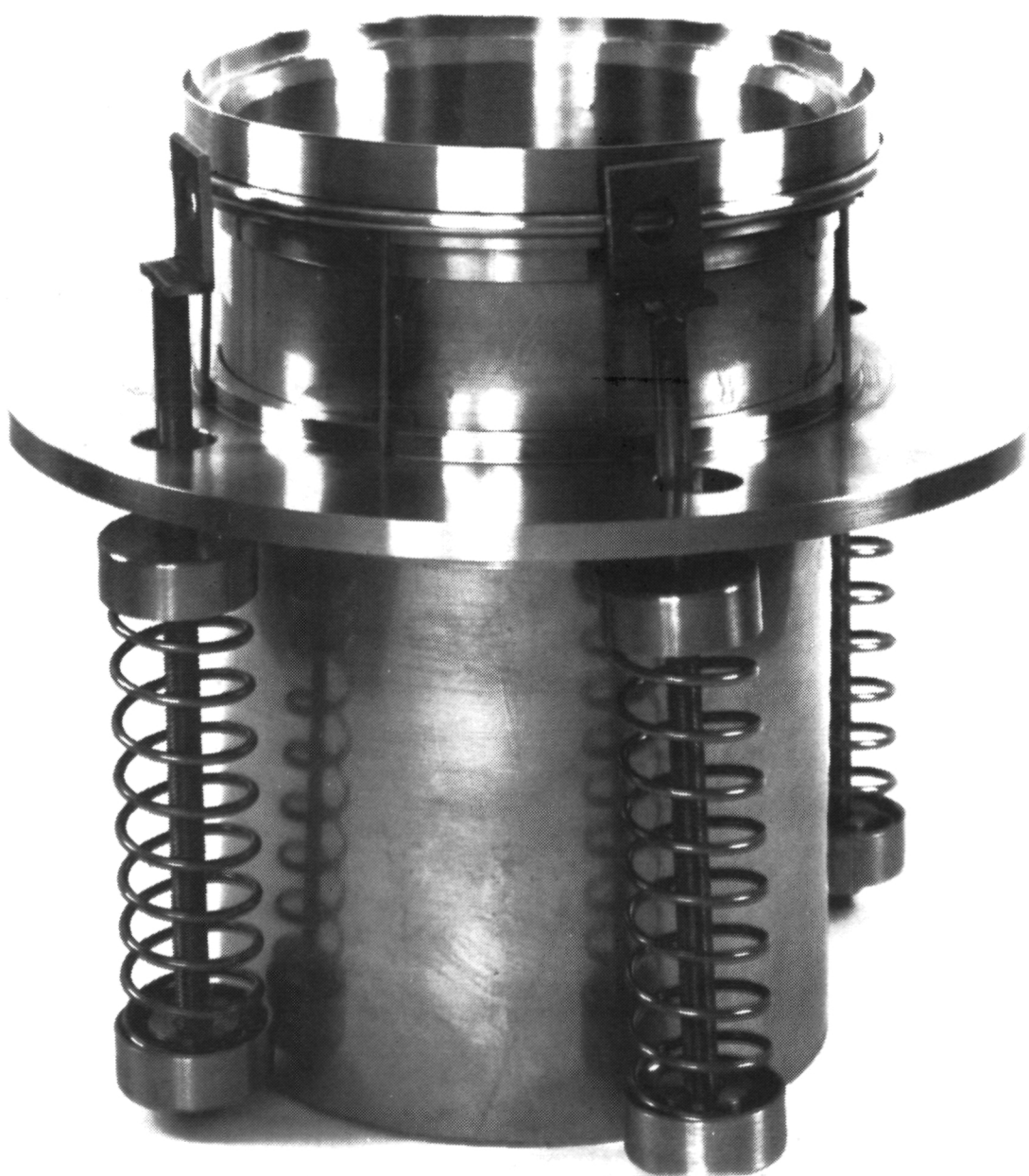
FIGURE 6.12   Quartz Liner Support for Free-Space Reactor PDU

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Experimental Runs (14 through 17)

Planned modifications to the free-space reactor PDU were completed and the unit was returned to operation in October 1980. Modifications included installation of a new vacuum line to the hopper. The vacuum pump was relocated to an outside shed and a 30-foot long, 2-inch diameter, copper line has been run to the PDU. Several instrument lines were also rerouted to eliminate conflicts with fluid-bed piping. Following a careful cleaning with deionized water, the free-space reactor was sealed; and after drying, purging, and leak detection, the PDU was started up on the 23rd of October, 1980.

Three consecutive runs (Runs 14, 15, and 16), accumulating a total of 11 hours, were made. Run 17 (12 hours duration) was made in November 1980. In all cases silane conversion exceeded 99.99% (no silane could be detected in the effluent). In all three tests no operational problems were encountered. Inspection of the hopper and reactor with a borescope showed the liner to be intact. The new powder transfer system was operating efficiently, much better than the older system. Following shutdown and purging, the powder was transferred and stored under a dry nitrogen blanket to avoid air and moisture contact.

The powder remaining in the hopper was vacuumed into drums and weighed. A mass balance around the PDU for runs 14 through 17 was then calculated:

	SiH_4	Si Produced	Si Recovered	H_2 Produced	H_2 Measured
Runs 14-17	53.75 kg	47.00 kg	39.05 kg	6.75 kg	7.05 kg (104.4%)
"Post Operation"			7.81 kg		
Total			46.86 kg (99.7%)		

The silane consumption was determined by weighing the silane cylinders and the silicon and hydrogen produced were then calculated from this figure. The hydrogen flow was measured with a dry test meter. A systematic error in estimating purge flows is probable as the measured hydrogen flow exceeded the calculated flow by approximately the same amount for each run. The "post-campaign" silicon recovered is the silicon removed by vacuuming after the system was opened. The silicon balance is excellent and the hydrogen balance acceptable.

Analytical results for Runs 14 - 17 are summarized in Table IX. The results of the purity analyses are comparable with those of previous runs. It was very clear that standard analytical methods were not adequate for our purpose. Melting of the powder followed by pulling of a single crystal and analysis of chemical composition appeared to be the only procedure capable of determining the suitability of free-space reactor silicon for solar applications. The results of the BET tests are of interest. Although the two samples shown are not significantly different in particle size, they both differ dramatically from the powder produced in Run 3 which had a mass average particle size of 0.92 μm. The very large increase in temperature in the upper section of the reactor caused by modifications made to the PDU during Phase II operations is undoubtedly responsible for the dramatic decrease in particle size.

TABLE IX

FSR PDU ANALYTICAL SUMMARIES

Run No.	Date	SiH$_4$ Rate (kg/hr)	Particle[1] Size	Ni	Purity (PPM)[2] Cr	Mn	Fe
14	10-23-80	2.30		0.9	0.20	0.08	9.0
15	10-28-80	2.33	0.34 m	0.48	0.60	0.25	22.0
16	10-31-80	2.29	0.34 m	1.4	0.15	0.10	11.0
17	11-10-80	2.37		1.3	1.0	0.15	13.0
Polysilicon Control				0.10	0.76	0.50	3.3

1   Based on BET surface area. Assumes spherical particles

2   Atomic absorption (Fe using flame; Cr, Mn, Ni using graphite furnace)

Final Test Run (Number 18, Long Duration)

The 12-hour duration run was completed on December 22, 1980. This run was made using the new scraper seal and was very smooth. Peak reaction zone temperature was 880°C, with a silane flow of 2.4 kg/hr; again silane conversion was better than 99.99%.

Other changes made to the PDU for Run 18 were the addition of an extra insulation collar above the main insulating blanket and the removal of 3 inches of insulation from the bottom of the reactor. The effect of these changes was to steepen the temperature gradient in the upper section of the reactor, flatten the temperature peaks in the mid-section of the reactor, and start the sharp drop-off in temperature closer to the top of the reactor. This profile is better than that obtained in earlier runs, see Figure 6.13.

Run 18 was the first test run to utilize the new scraper (see Figure 6.14). This scraper differs from that used in previous runs in that it uses an in-line double-ended air cylinder instead of a side-mounted single-ended cylinder, and separate elastomeric wipers and 0-ring seals instead of simple packing rings. The new scraper should require less maintenance than the original unit. It also has a longer stroke - 0.75 m vs 0.45 m in the older scraper. Performance of the new scraper during Run 18 was smooth. The unit will be inspected for wear as soon as the unit is opened.

Run 18 was the last run called for in the FSR PDU program plan. Thus, all the runs planned for this phase were successfully completed and have demonstrated the operational feasibility of the system.

Table X gives the conditions for runs 14 through 19. (Run 19 was made on the last day of the year, to gain the maximum time of operation and terminated after 3.7 hours, as a result of silane depletion.)

Powder Inventory and Shipping

Kayex Corporation of Rochester, New York (subcontractor for silicon melter system development) fabricated a powder transport drum for powder

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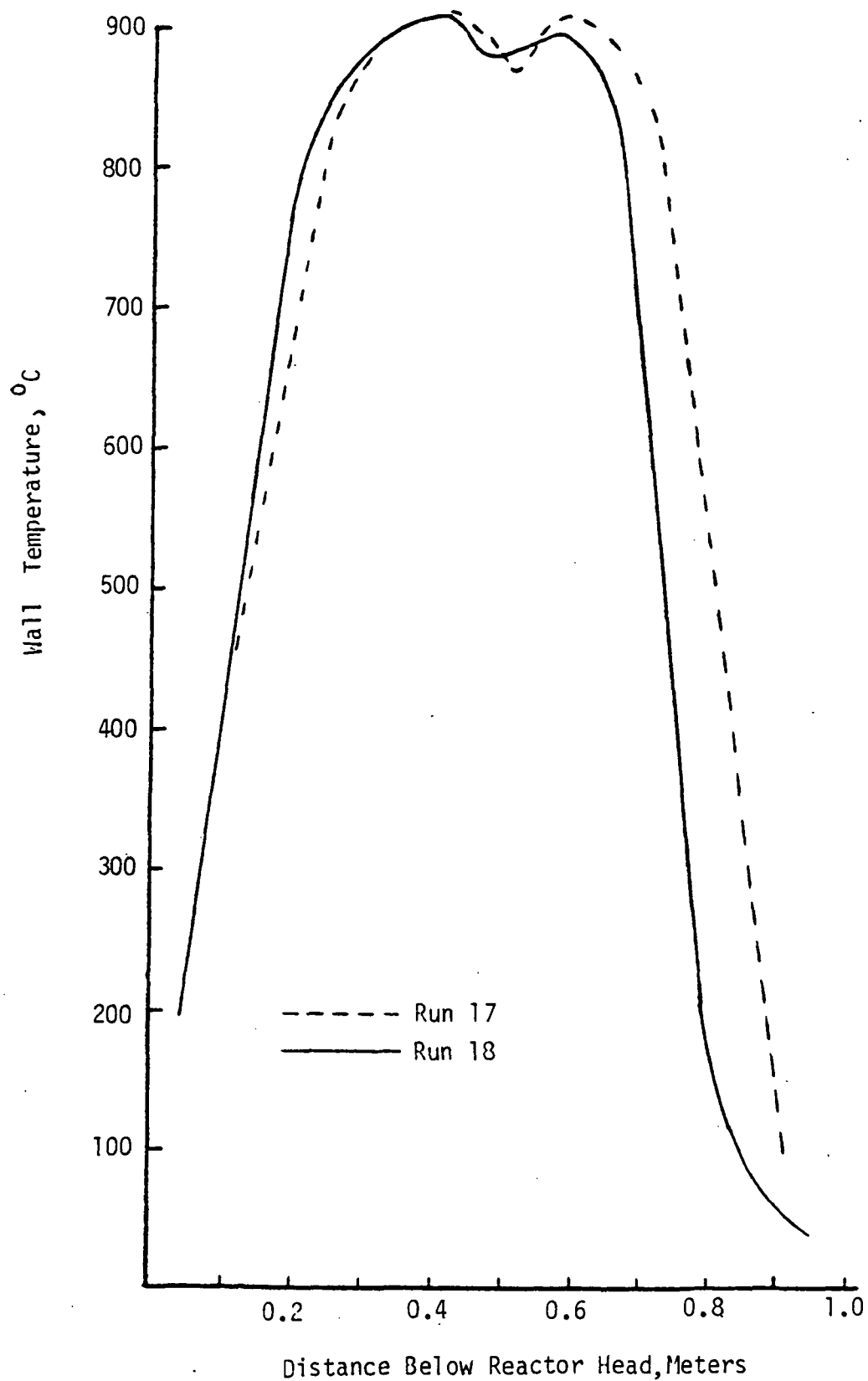
Figure 6.13   FSR-PDU Wall Temperature Profile

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PNEUMATIC CYLINDER
(WITH MAIN O-RING SEALS)

UPPER PURGE
(PRESSURIZED, FLOW-THROUGH)

PURCHASED SEAL/BEARING ASSEMBLY

CONNECTION FOR ALTERNATE
SEAL ASSEMBLY (BELLOWS)

ADAPTER

LOWER PURGE
(BLEED INTO REACTOR)

LOWER BEARINGS

LANTERN RING

LOWER WIPER

WATER COOLING

REACTOR HEAD

SHAFT
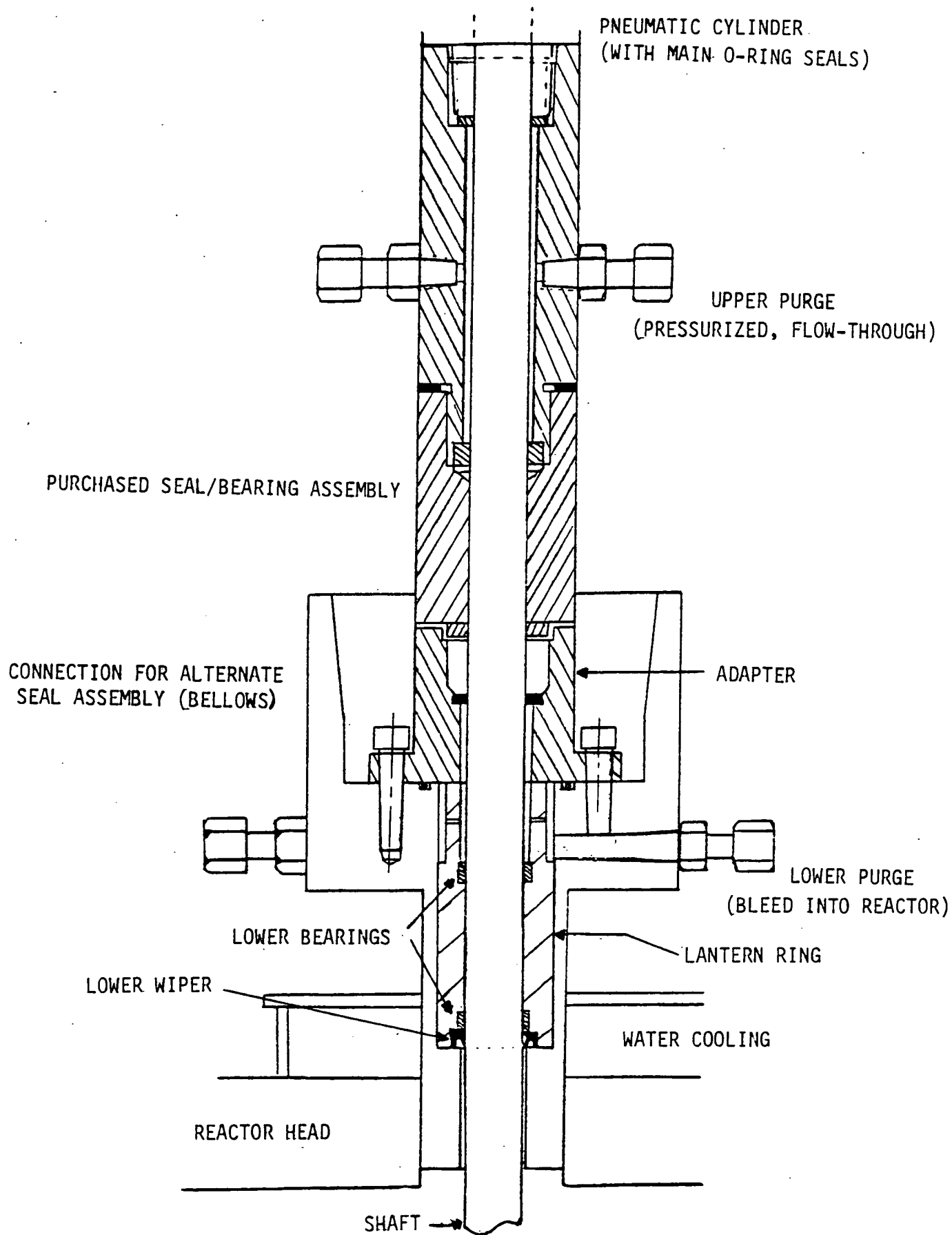
FIGURE 6.14   FSR New Scraper Seal

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TABLE X RUN SUMMARIES (14-19)

Run No.	Date	Duration (Hrs)	SiH$_4$ Rate (kg/hr)	Reaction Zone Temp (^{o}C)	Powder Transferred (%)	Comments - Equipment Modification
14	10/23/80	3.0	2.30	865	75	New liner support and powder transport systems installed. Heating system unchanged. Powder stored under inert purge.
15	10/28/80	4.0	2.33	875	83	As in Run 14.
16	10/31/80	4.0	2.29	860	101	Completion of milestone. As in Run 14.
17	11/10/80	12.0	2.37	870	79	Completion of milestone. As in Run 14. 11.8 kg to Kayex.
18	12/22/80	12.0	2.4	880	57	New scraper installed. Other systems unchanged.
19	12/31/80	3.7	2.3	880	75	Consumption of last available silane (remaining cylinder pressures too low). Minimal effort to transfer powder.

shipments. This consisted of a modified stainless steel 55-gallon drum incorporating a screw auger, breaker bar, and porous fluidizing surfaces. The drum was used successfully by Kayex to transfer the powder to a melter crucible without exposure to the atmosphere.

Approximately 100 kg of silicon powder was required for shotting work by Kayex. The powder inventory (December 30, 1980) was 50 kg of good powder kept under nitrogen purge, and approximately 50 kg of powder with silicon chunks which had to be screened out. We, thus, had roughly 100 kg of powder for shotting work and no surplus in storage.

Crystal Growth Experiment Using Free-Space Pyrolysis Powder

A crystal growth experiment was conducted by Kayex Corporation on October 20, 1980, using silicon powder produced by the free-space reactor. The primary objective was to establish the feasibility of growing an ingot by introducing silicon powder directly into the crystal grower.

A powder-feeding device based on an auger was designed and fabricated to transfer powder directly from a storage drum to the crystal grower. A CG 2000 RC puller was used. The drum-auger assembly, containing silicon powder from free-space reactor Run 13, was connected to an inclined quartz tube entering the crystal grower through a top port. A ball valve was used to isolate the crystal grower. A ceramic-disk plunger was provided to clear the quartz tube in case of blockage.

Powder was fed to the crucible by manually rotating the auger after applying power to the puller. Powder feeding was satisfactory, however, considerable blowing of the powder in the furnace chamber was observed. This uncontrolled blowing of fine powder caused arcing problems whenever the powder got in between the crucible and the graphite heating elements. Powder melting was effective. Since the power input had to be limited to 50 to 60 kw due to arcing, and the crucible could not be lowered into a higher temperature zone, the feeding and melting steps were rather slow, taking almost 6 hours to establish a stable melt of approximately 6.5 kg of

silicon. The melt was relatively free from silicon monoxide; however, a glassy slag formation observed in the melt was not explained until completion of the experiment. When the equipment was disassembled at the end of the run, it was realized that the ceramic plunger disk (1.25" dia. x 0.25" thick, containing approximately 7% boron) (designed by Kayex) had accidentally fallen into the melt.

Crystal pulling was hampered by several factors. Crucible rotation had to be stopped due to arcing problems. Fluxing and devitrification of quartz crucible, caused by the dissolved ceramic disk, resulted in quartz particles migrating towards the seed and affecting crystal growth. Kayex was able to pull polycrystalline ingots of 1 inch and 4 inches in diameter and 3 inches long. Due to the heavy accidental doping, resistivity of the poly-silicon ingots was low, typically 0.04 Ω cm, P-type.

On November 20, 1980, a second powder-melting and crystal-pulling run was conducted by Kayex Corporation using silicon powder produced by the free-space reactor. The powder from our Run No. 17 was transferred into the stainless-steel transfer-drum directly without exposure to the atmosphere. The drum was initially kept under a nitrogen purge and finally under an argon purge. This drum, containing an auger, was connected to Kayex's CG 2000 RC crystal puller. The ceramic plunger, which caused contamination problems in the previous run, was eliminated. The crystal-puller furnace was equipped with a new graphite hot zone. A quartz ring was used to shield the annular space between the quartz crucible and graphite heater. This shield prevented powder from getting into the annular region and thus eliminated arcing prob-lems experienced during the previous run.

Powder feeding and melting operations were effective. The feed and melt rate was approximately 3.2 kg/hr, and there were indications that this rate could be considerably increased. Attempts to pull a single crystal ingot were hampered by the presence of a contaminant "scum" floating on the melt surface. This contamination seemed to originate from powder that had reacted with some component of the furnace atmosphere. When the gas flow around the crucible was disturbed during attempts to remove the contaminants,

additional powder with contaminant fell into the melt. The bulk powder did not appear to contribute to this contamination.

Kayex was able to pull a polycrystalline ingot 3 inches in diameter x 4 inches long. The resistivity of this poly was measured to be 55 Ω cm, P-type.

Reactor Scale-Up and Design

A 100 MT/year free-space reactor/silicon hopper assembly for EPSDU was designed based on the experience gained in operation of the PDU during the later half of 1980 and a theoretical analysis.

The design consists of a free-space reactor, resistance heating furnace, and product hopper. Detailed design drawings for fabrication of the equipment were prepared, and the calculations were documented in a separate design calculation package. The reactor and hopper were designed in accordance with established engineering practice for pressure vessels with ASME-code formulae serving as a design basis. Both the reactor and the hopper were designed for a maximum allowable working pressure (MAWP) of 14.0 psig with design temperatures of $1000^{\circ}C$ for the reactor shell, $300^{\circ}C$ for the reactor head, and $500^{\circ}C$ for the hopper. Additionally, both the reactor and hopper are capable of withstanding full vacuum.

The reactor consists of a cylindrical Incoloy 800H shell, an 18-inch I.D. quartz liner, and a water-cooled stainless steel top head. The hopper is constructed of 304 stainless steel and consists of torispherical upper head, a 48-inch O.D. cylindrical main shell section with an 18-inch access manway, and a Dynapore (porous 304 stainless steel) lined lower conical shell. These components are shown in Figure 6.15.

Silane gas is injected into the quartz-lined reaction chamber via a water-cooled injector tube which passes through the reactor head. The optimum temperature profile in the reactor is maintained by an externally

USED ON ASSEMBLY NO.

TOLERANCES UNLESS OTHERWISE NOTED
MACHINED SURFACES SHALL BE

U/M	ITEM NO	LINDE PART NO CODE NO	REG FOR ONE	MATERIAL AND DESCRIPTION	DWG ALT LETT

FRONT ℄

302.50 (25'-2.50") CYLINDER IN DOWN POSITION
342.50 (28'-6.50") CYLINDER IN UP POSITION

84.00
REACTOR

167.75 (13'-11.75)
HOPPER

71.00

1.77.25

112.00

SD-3

SD-2

20.75 DIA. REF.

48.00 DIA. REF.

46.00 REF.

54.00

SEE NOTE 3

21
22
23

SEE NOTE 2

SD-1

74.25

FIGURE 6.15

FOR NOTES BILL OF MATERIAL & SD's
SEE BM-2160600

REFERENCE DRAWINGS
DESIGN CALCULATIONS   A-2160633
CONNECTION LAYOUT   C-2160643

WARNING MUST BE CLEANED PER LINDE
STANDARD SPEC. SCS-1

TITLE: FINAL ASSEMBLY
FREE SPACE SILICON REACTOR /
SILICON HOPPER

	BY GS	DATE 3/5/81	SCALE 1"=1'-0"
CHECKED Jay			
REVIEWED SCS			
APPROVED Jay			

UNION CARBIDE CORPORATION
LINDE DIVISION
TONAWANDA NEW YORK

D-2160600

ESTIMATED WEIGHT

HOPPER & REACTOR ASSEMBLY — 5225 LBS
FURNACE ASSEMBLY — 4000 LBS

LIST ALTERATIONS ON BM-2160600 GR. I

ALTERATION | DATE | BY | CH'K'D | APPV'D | LETT

mounted multi-zone resistance furnace with provisions for feedback temperature control. The furnace is supported independently of the reactor/hopper vessel by two I-beams which attach to an external framework.

During the process, silane gas (SiH_4) is decomposed to form silicon powder (Si) and hydrogen gas (H_2). The reaction products enter the hopper via a 20-inch communicating chamber in the upper head of the hopper. The silicon powder collects in the lower conical and straight shell sections of the hopper.

A manifold, consisting of eighteen sintered metal filter elements, is attached internally to the upper head of the hopper with a 2-inch pipe size nozzle passing through the head. Hydrogen gas from the reaction is drawn from the hopper through the filters to a separate external recovery system. The hopper has a net product volume of 79 cubic feet to a point 12 inches below the filters.

A product withdrawal line (1-1/2 inch diameter pipe) passes through the main shell of the hopper and terminates near the bottom of the conical shell. Hydrogen gas is injected from an external source through the lower cone flange, into the annular space between the outer shell and the Dynapore (porous stainless) inner cone. The flow of gas facilitates pneumatic transfer of the powder from the hopper through the product withdrawal line. Powder flow is assisted by the flow of hydrogen gas through the sintered metal cone, and by an internal "breaker bar" mechanism mounted through the main shell immediately above the conical shell juncture. The "breaker bar" mechanism consists of 0.5-inch stainless steel rods (adjustable) mounted to a 1-inch stainless steel shaft which is driven by an externally mounted pneumatic rotary torque actuator to break up powder "bridging" in the hopper shell and cone.

Powder deposits on the quartz reactor chamber walls are removed by a stainless steel "scraper" ring capable of traversing a 40-inch length of the reactor chamber. The scraper is mounted on the end of the water-cooled shaft of a specially-designed (Carter Controls) pneumatic cylinder suitable for high temperature operation. The pneumatic cylinder is externally attached to the top of the reactor head. The cylinder shaft passes through the water-

cooled reactor head which contains an additional scraper and sweep purge chamber to protect and insure proper functioning of the pneumatic cylinder.

Cooling for the hopper is provided by clamp-on Panelcoil elements attached to the cylindrical shell of the hopper and by 0.5-inch O.D. copper tubing coils on the upper hopper head. Additional copper cooling coils are attached to the lower portion of the reactor shell (below the furnace). Heat transfer to the cooling coils and panels is enhanced by the use of a heat transfer cement.

The reactor/hopper assembly is supported by four external lugs attached to the main shell of the hopper. The support lugs will in turn be bolted to the same external framework to which the independently supported furnace is attached.

Drawing number D-2160600 (Figure 6.15) shows the final assembly — Free-Space Silicon Reactor/Silicon Hopper for the EPSDU facility.

The EPSDU Free-Space Reactor was designed to process a maximum silane throughput of 75 lb/hr at a maximum wall temperature of $925^{\circ}C$. This throughput, which represents a rate about 50% higher than the steady-state production rate required to produce 100 metric tons of silicon per year, allows for plant downtime.

Computer simulations of the free-space reactor predicted that an 18-inch diameter reactor will be capable of successfully reacting 75 lb/hr of silane without hard wall deposit formation. On this basis, the preliminary design calls for the reactor liner to have an 18-inch inner diameter. The reactor shell, which encloses the quartz liner, will be constructed from Incoloy 800H and will have the following dimenstions: 20-inch O.D., 6-foot length, and 5/8-inch wall thickness. It is designed to withstand pressures up to 50 psig at temperatures up to $1000^{\circ}C$. At the normal operating pressure of 17 psig, the stresses in the reactor cylinder are 225 psi, which is much less than the 700-psi rupture stress of Incoloy at $950^{\circ}C$.

A multizone resistance heating system with automatic feedback temperature control will be used for the EPSDU reactor instead of an induction heating system such as that used in the PDU. Experience from the PDU indicated that induction heating has two major drawbacks in this application.

- low heating efficiency resulting mainly from high power losses due to inductive coupling with metal surfaces near the long power leads.

- the fixed temperature profile obtainable from a given coil.

A properly insulated resistance heating system should have lower heat losses, and multizoning of the heating elements allows for flexibility in establishing the desired temperature profiles.

Curved resistance heating elements with a 45° arc and 6-inch width, a temperature capability of $1200^\circ C$, and a power rating of 1025 watts per unit are commercially available. The heater design calls for two semi-circular assemblies constructed of 48 heaters each, with four heater elements per axial band, and twelve axial bands spanning the length of the reactor. The two heater assemblies will be hinged together to allow access to the reactor cylinder and easy replacement of elements. Figures 6.16 and 6.17 illustrate the basic design concept.

A feedback control circuit monitors the wall temperature in a given zone with a thermocouple, and a temperature controller varies the amount of power supplied to the heaters in that zone to maintain the desired reactor wall temperature. The preliminary design calls for six controllers with each of the first three bands independently controlled (since this is the most critical region from a process standpoint), and the other three controllers controlling three bands each. The design is shown schematically in Figure 6.18. Reflective baffles reduce interaction between heaters in adjacent bands. A total of 12 independently-controlled zones can be created by adding additional control circuits.

Labels on figure:

AIR GAP BETWEEN REACTOR AND HEATING ELEMEN (2 ¼")

INCOLOY 800H REACTOR CYLINDER (20"OD, 6' long, 5/8" wall thickness)

QUARTZ LINER (18" ID, 3mm thickness)

RESISTANCE HEATING ELEMENTS (24½" ID, 6" length, 45° arc)

CERAFELT INSULATION (2" thick)

REFLECTIVE BAFFLES

6'

18"

20"

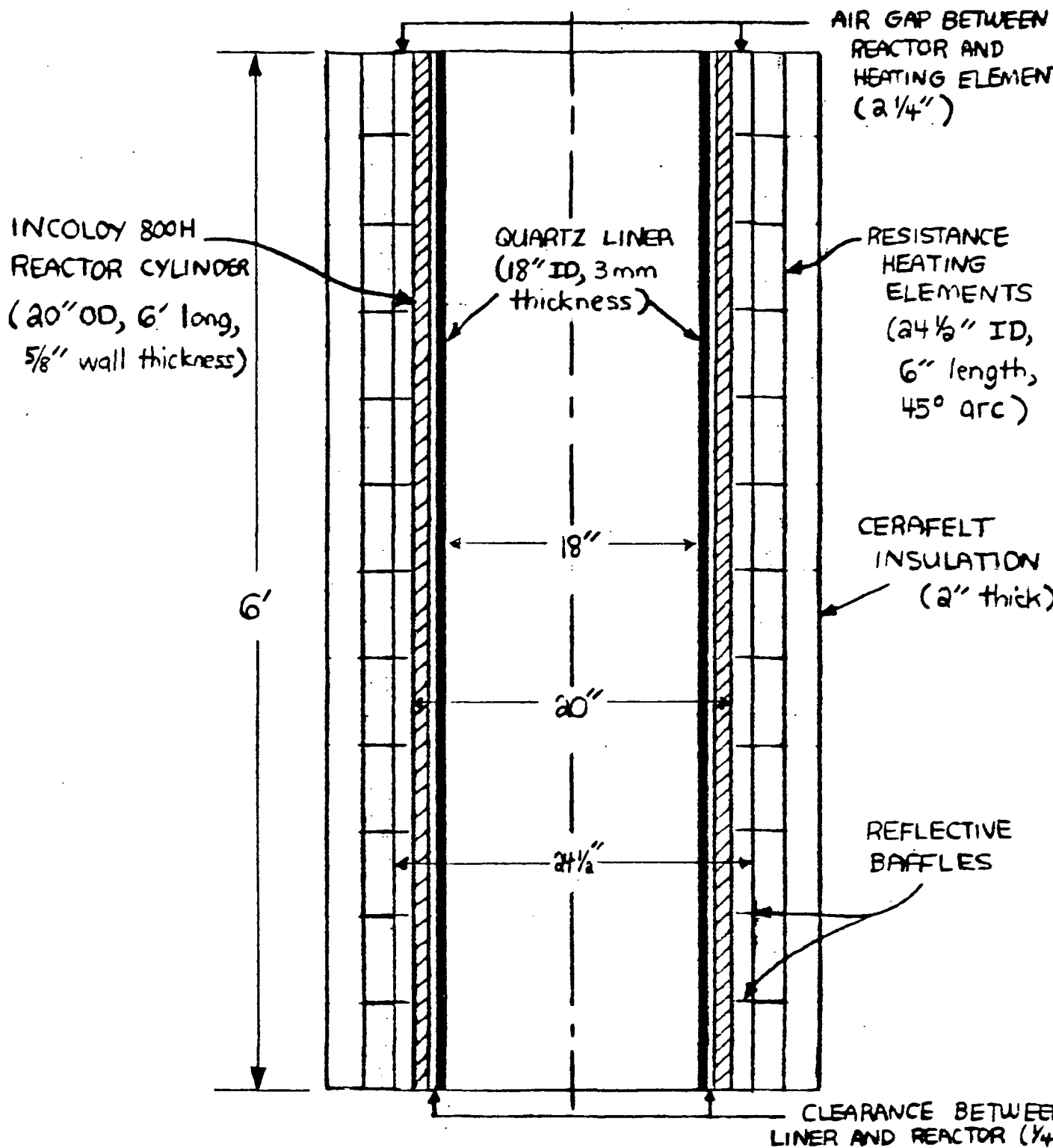24½"

CLEARANCE BETWEE LINER AND REACTOR (¼

FIGURE 6.16   PRELIMINARY CONCEPTUAL DESIGN OF EPSDU
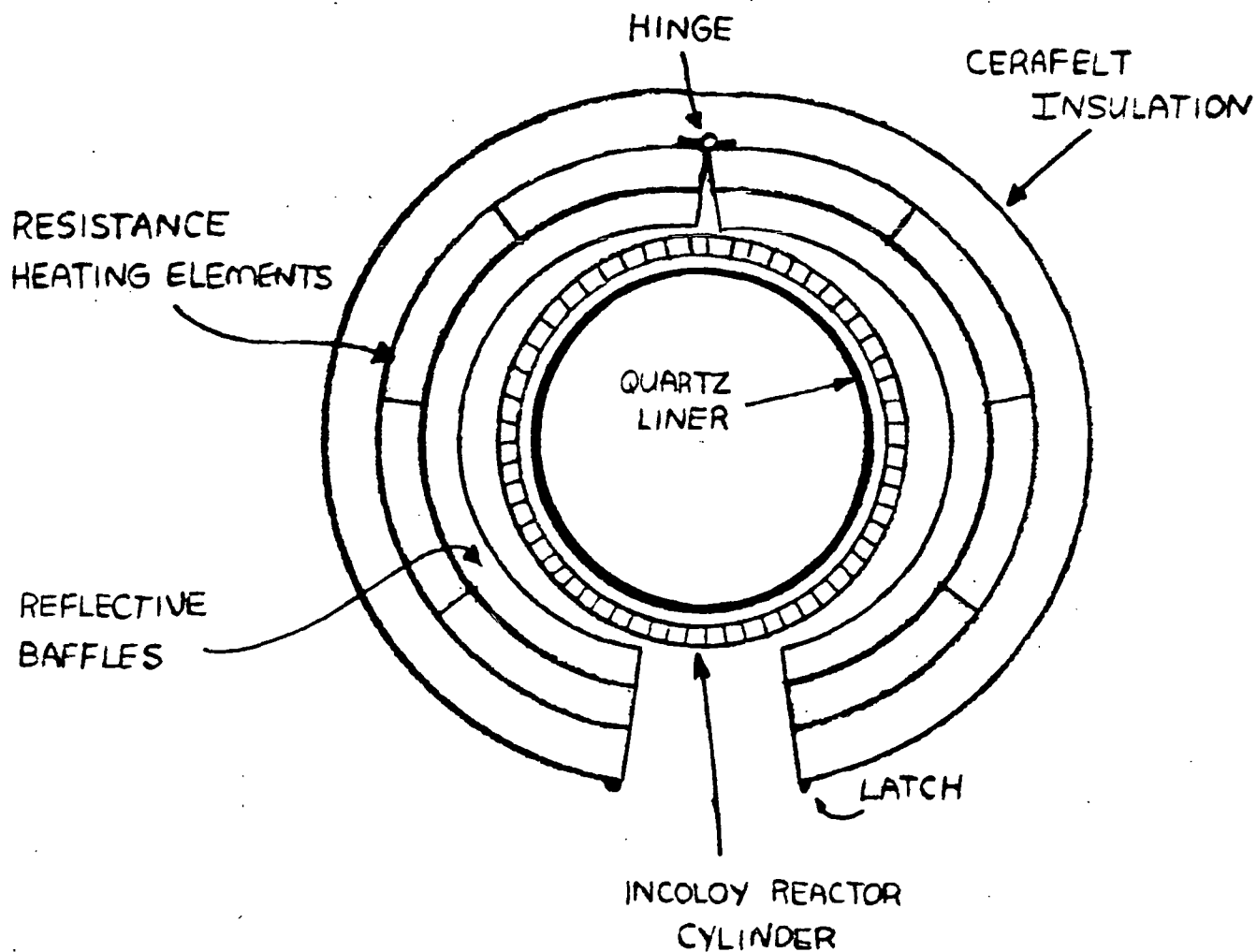FREE-SPACE REACTOR HOT ZONE - SIDE VIEW

FIGURE 6.17 PRELIMINARY CONCEPTUAL DESIGN OF EPSDU
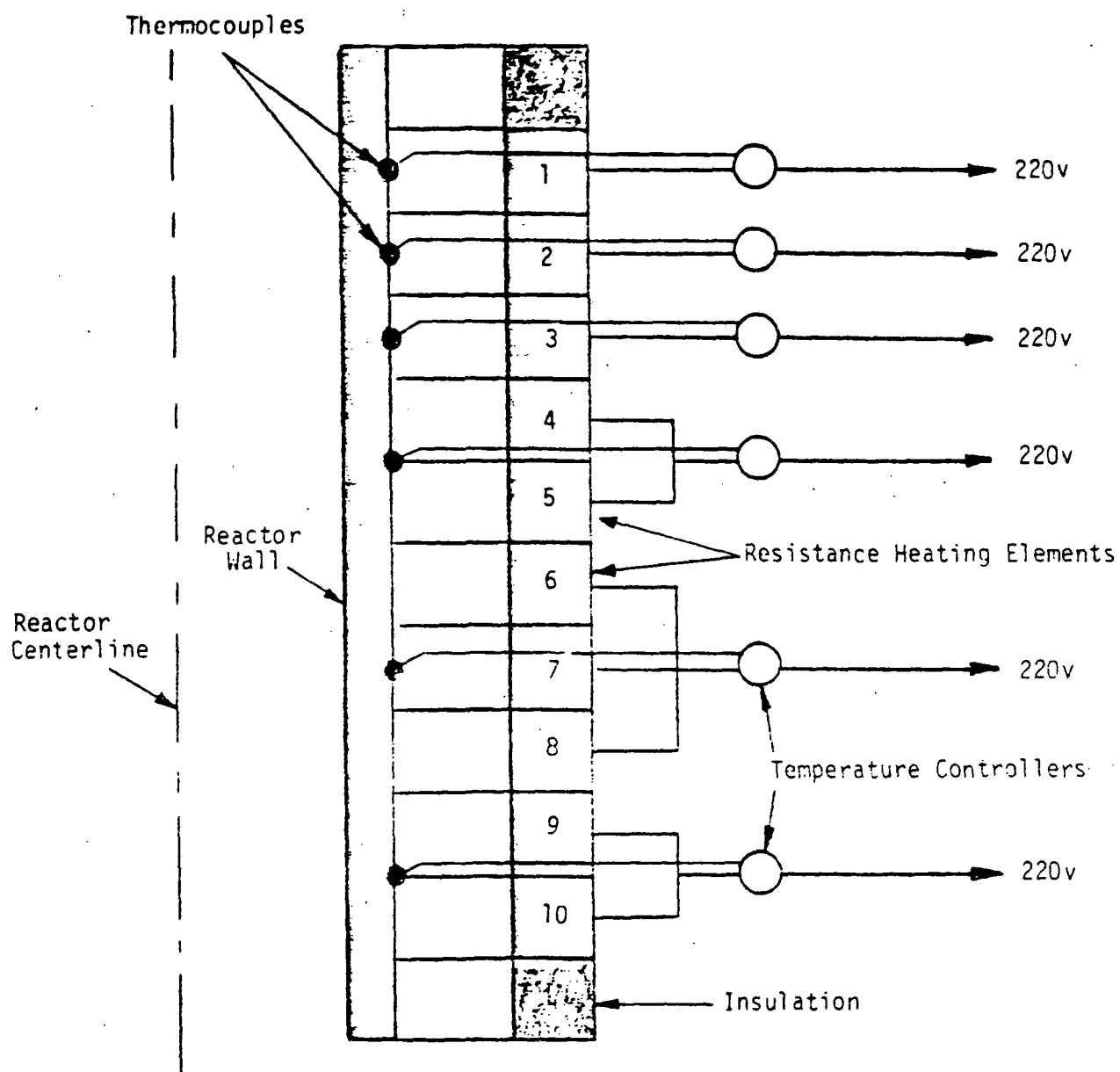FREE-SPACE REACTOR HOT ZONE - TOP VIEW

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FIGURE 6.18 Control System for EPSDU Multizone Resistance Heating System

The 96 heating elements give the heating system a capacity of almost 100 KW. Since the process requirements, including losses, are on the order of 50 KW, the system has considerable reserve capacity. Under normal operating conditions, the resistance elements will draw considerably less than their maximum power rating which will extend heater life. The system has sufficient power to heat the reactor to operating temperature in under 2 hours.

A computer model of gray body-radiation exchange between the resistance heaters and the reactor wall was developed to predict the temperatures and power inputs needed in the heating elements in the various zones to attain the required temperature profile in the reactor wall. Results predicted that the proposed system is capable of producing the desired temperature profile.

Analyses from this model showed conclusively that reflective baffles between the independently-controlled heating zones are an absolute necessity if the resistance heating system is to be capable of achieving the required temperature profile in the reactor wall. Without baffling, interaction between the heating zones becomes a serious problem. As the gap between the reactor wall and the heating elements becomes larger, interaction between the element becomes more and more significant, since the radiation view factors between adjacent zones increase. As the gap becomes larger, the required power input to the second zone increases until it exceeds the power rating of the heating elements. At the same time, the power input to the first zone becomes smaller until, eventually, heat actually flows out through the first zone. The temperatures in the second zone increase and those in the first zone decrease in the same manner as the gap is increased. Baffling of the various zones to reduce interaction will have a similar effect to reducing the gap to a very small distance, and the heat flux distribution in the resistance elements should approach that in the outer reactor wall.

A simulation run of the model was made to determine whether the proposed EPSDU resistance heating system was capable of generating the wall temperature profile required in the EPSDU reactor. The reflectivity of the

ceramic baffles and the ceramic backing of the heating elements was assumed to be 0.5, and the reactor wall was assumed to be black. A 2-inch gap between the heating elements and the reactor wall was specified.

Figure 6.19 shows the power inputs to the various heating zones, and Figure 6.20 shows the corresponding element temperatures needed to achieve the desired temperature profile. The maximum power input and element temperature was predicted in Zone 3, with values of 3.4 KW and 994°C respectively. Since each zone of heating elements has a power rating of 8 KW and a temperature capability of 1200°C, the system was capable of achieving the desired profile. The negative power inputs to Zones 1 and 12 indicate that heating elements are unnecessary in these regions provided these areas are properly insulated.

1.6.1.2   Theoretical Analysis

The purpose of the theoretical analysis was to develop a mathematical model of the free-space reactor that could be used to determine optimal values for the reactor operating parameters, and assist in scale-up and design of the EPSDU prototype reactor.

The early portion of the theoretical effort consisted of a detailed evaluation of previous models of the free-space reactor developed by other investigators. This evaluation indicated the unsuitability of the previous models in meeting the objectives of the theoretical program. Effort was then directed at developing a two-dimensional axisymmetric model capable of analyzing the recirculating flow in the reactor.

The mathematical model of the free-space reactor that was developed treats the reactor as a compressible, turbulent, axisymmetric, chemically-reacting confined jet. The conservation equations for momentum, mass, energy, and species are formulated for the reactor, and solved numerically by a control volume finite difference procedure. The conservation equations represent a mathematical description of the fluid mechanics, heat transfer, and chemical reaction in the reactor. The quantitative validity of this model has been
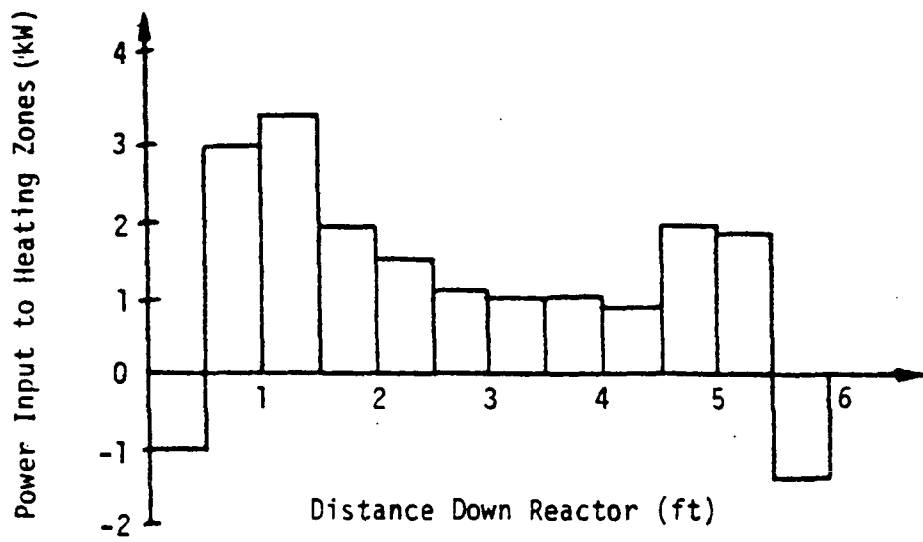
FIGURE 6.19 Power Inputs to the Heating Zones -
EPSDU Reactor Simulation
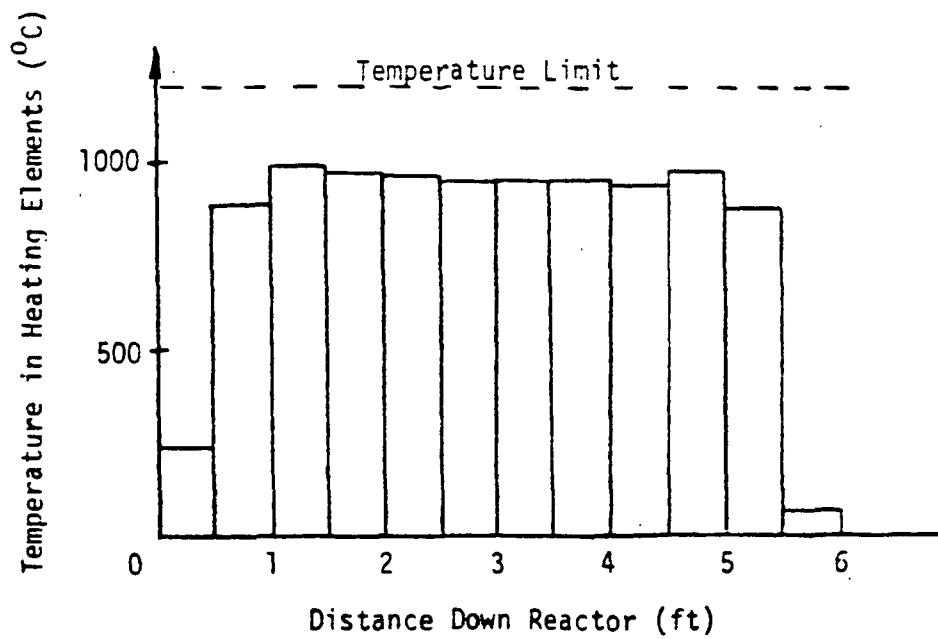
FIGURE 6.20 Temperatures of the Heating Elements -
EPSDU Reactor Simulation

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established by extensive comparison of experimental results and model predictions. The experimentally-verified model serves as a rational basis for scale-up of the free-space reactor from the current PDU to an EPSDU sized unit capable of producing 100 metric tons of silicon per year.

The mathematical model of the free-space reactor consisted of the following:

- Conservation equations for momentum, mass, energy, and species along with boundary conditions appropriate to the reactor configuration.

- A turbulence model that accounts for the effects of turbulence on the velocity and temperature fields.

- A reaction model that accounts for the depletion of silane by the reaction and the resultant heat release.

The flow in the free-space reactor is assumed to be axisymmetric. The conservation equations are written in cylindrical coordinates, and form a non-linear coupled system of partial differential equations.

The dependent variables of interest in the free-space reactor are the axial velocity, the radial velocity, the pressure, the temperature, and the silane mass fraction. Due to the turbulent nature of the flow, the conservation equations are formulated in terms of the mean-time-average values of these variables. The effects of the turbulence are accounted for by the K-ϵ turbulence model.

The numerical solution procedure used in the model is the control-volume finite difference procedure pioneered by Spaulding's group at Imperial College in London. This numerical procedure appears to be one of the most suitable methods currently available for solving complex problems involving heat and mass transfer in recirculating flows. The computer program FREE-SPACE that is used to solve the equations describing the free-space reactor is an extension of the TEACH program developed at Imperial College.

In cylindrical polar coordinates, the conservation equations can all be written in a standard elliptic form. For steady flow the standard form is:

$$\frac{\partial}{\partial x}(\rho u \emptyset) + \frac{1}{r}\frac{\partial}{\partial r}(r\rho V \emptyset) - \frac{\partial}{\partial x}\left(\Gamma_{\emptyset}\frac{\partial \emptyset}{\partial x}\right) - \frac{1}{r}\frac{\partial}{\partial r}\left(r\Gamma_{\emptyset}\frac{\partial \emptyset}{\partial r}\right) = S_{\emptyset}$$

Here $\emptyset$ stands for the time-average value of any of the dependent variables (U, V, P, T, M_s, K, ε), $\Gamma_{\emptyset}$ is the appropriate diffusion coefficient for the variable $\emptyset$, and $S_{\emptyset}$ represents source terms associated with the equation of $\emptyset$.

The numerical solution procedure solves the conservation equations in their standard form. Finite difference equations at a grid point are obtained by integrating the governing differential equations over a control volume centered about the point. Diffusion fluxes across a control volume face are computed assuming a linear variation of the flow properties between adjacent grid points. Convective fluxes are computed using a hybrid differencing scheme that uses either an upwind or central difference depending on the local grid cell Peclet number. Formulation of the finite difference equations in this manner guarantees that even coarse grid solutions to the equations are physically realistic and satisfy overall conservation exactly.

The resulting finite difference equations are of the form:

$$A_p \emptyset_p = A_n \emptyset_n + A_s \emptyset_s + A_e \emptyset_e + A_w \emptyset_w + S_{\emptyset}$$

The system of equations is solved iteratively by a tri-diagonal matrix algorithm. The staggered grid system and SIMPLE algorithm of Patankar and Spaulding is used to solve for the velocity and pressure fields. Further information on the numerical method is available in many references.

Experimental Verification of Model

Detailed measurements of the velocity, concentration, and temperature fields inside the free-space reactor were beyond the scope of the

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experimental program. Tests on the validity of the theoretical model were therefore limited to comparisons of quantities that were measured experimentally, and trends that were observed. Excellent qualitative agreement of the model with experimental trends due to changes in silane throughput and reactor wall temperature was observed. The quantitative validity of the model was established by the following criteria:

- Quantitative agreement with internal temperature measurements made during early experiments at Parma.

- Quantitative agreement with throughput capacity tests conducted at Tonawanda.

- Ability to predict operating conditions leading to hard wall deposit formation.

Internal Temperature Comparison

Obtaining accurate measurements of internal temperatures during reactor operation is extremely difficult because the thermocouples quickly become covered with silicon powder and vapor deposited silicon. The only time that internal temperature measurements were made during actual reactor operation was at Parma during August 1977.

Figure 6.21 compares measured internal temperatures with the temperatures computed by the model for a typical experiment. The wall temperature profile measured experimentally was parabolic in shape, and is included in the figure for reference. The experimental profile was used in the model simulation. The predicted temperatures agree favorably with the measured values throughout the length of the reactor.

Throughput Capacity Comparison

A series of short term capacity experiments was conducted during the first phase of the experimental program at Tonawanda. The conversion efficiency as a function of silane throughput was determined experimentally by measuring the fraction of silane in the off gas with a gas chromatograph. The original uniformly-spaced coil that generated a parabolic wall temperature

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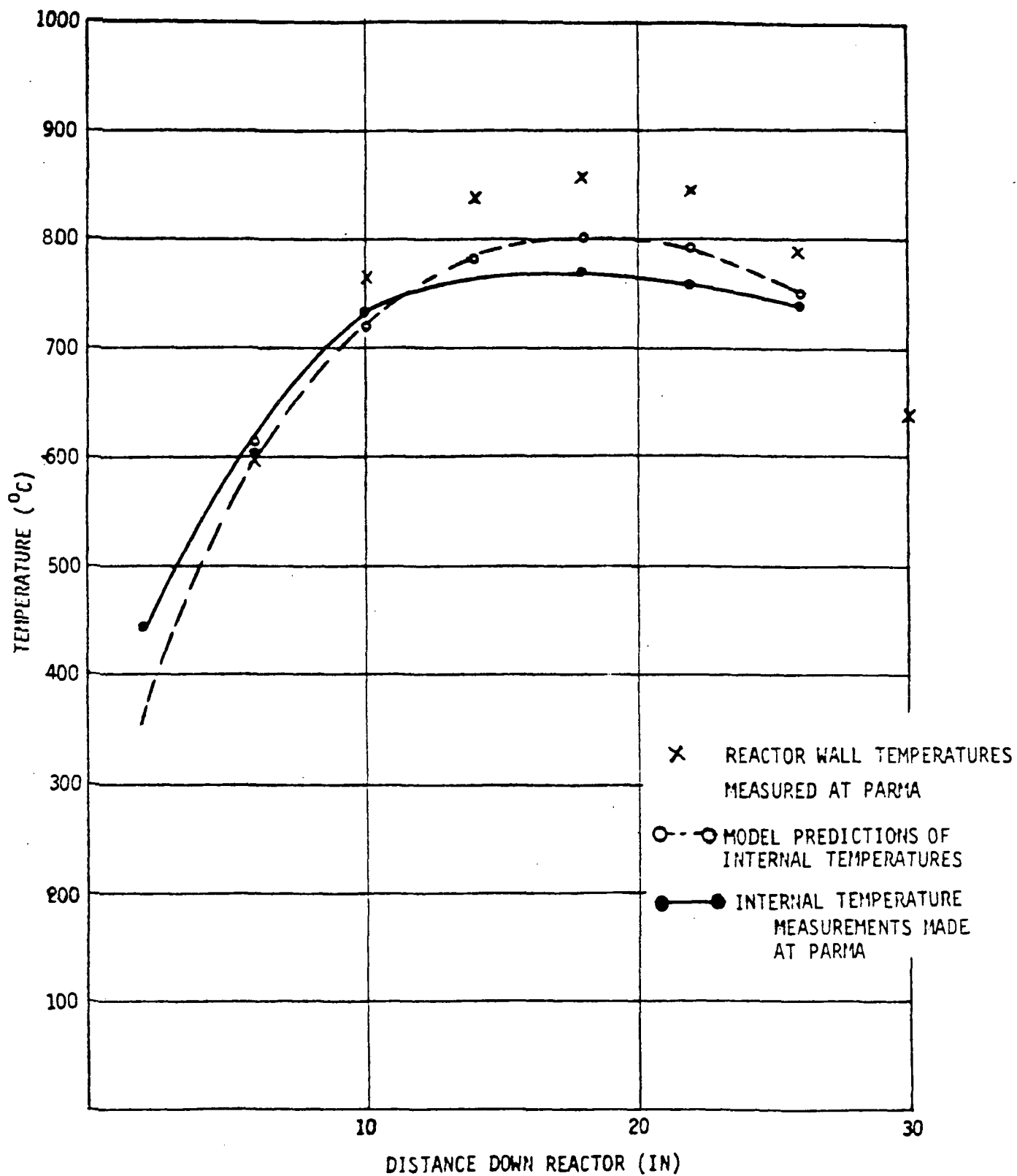
FIGURE 6.21 Internal Temperature Comparison

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distribution was used during these experiments. Reaction conversion effi-
ciencies in excess of 99% were measured at silane throughputs of up to
10 lbs/hr. The measured conversion fell to 85% at a throughput of 13.7 lb/hr.

The dramatic effect of the shape of the wall temperature profile
on the reactor operation became apparent when the model was used to simulate
these capacity experiments. The reaction conversion as a function of silane
throughput was predicted for two different wall temperature profiles:

- a uniform 950°C wall temperature, and

- a parabolic profile with a peak temperature of 950°C

The results, shown in Figure 6.22 show that the decrease in reaction with
increasing throughput is far more dramatic when the temperature distribution
is parabolic. In addition, for the same maximum wall temperature, the reac-
tion efficiencies are invariably higher when the wall temperature is uniform.

Prediction of Hard Wall Deposits

Experiments conducted with the PDU clearly demonstrated that the
throughput capacity of the free-space reactor is limited not by the require-
ment of high reaction efficiency but rather by the necessity of preventing
the formation of hard wall deposits. In the early experimental phase, ex-
cessive hard wall deposit formation led to reactor liner breakage even though
the measured reaction efficiencies were in excess of 99%. The success of the
model in predicting hard wall deposit formation is of the utmost importance
since the required size of the EPSDU reactor is determined from this criterion.

Heterogeneous decomposition of silane on the reactor wall cannot
occur if unreacted silane does not reach the reactor wall. By completely
reacting the entering silane before the expanding jet reaches the wall, hard
wall deposit formation can be eliminated. For any set of operating condi-
tions, the model predicts the location of the homogeneous reaction (by means
of the reaction intensity distribution function) and the resulting silane
concentrations that exist near the wall. This information, in conjunction
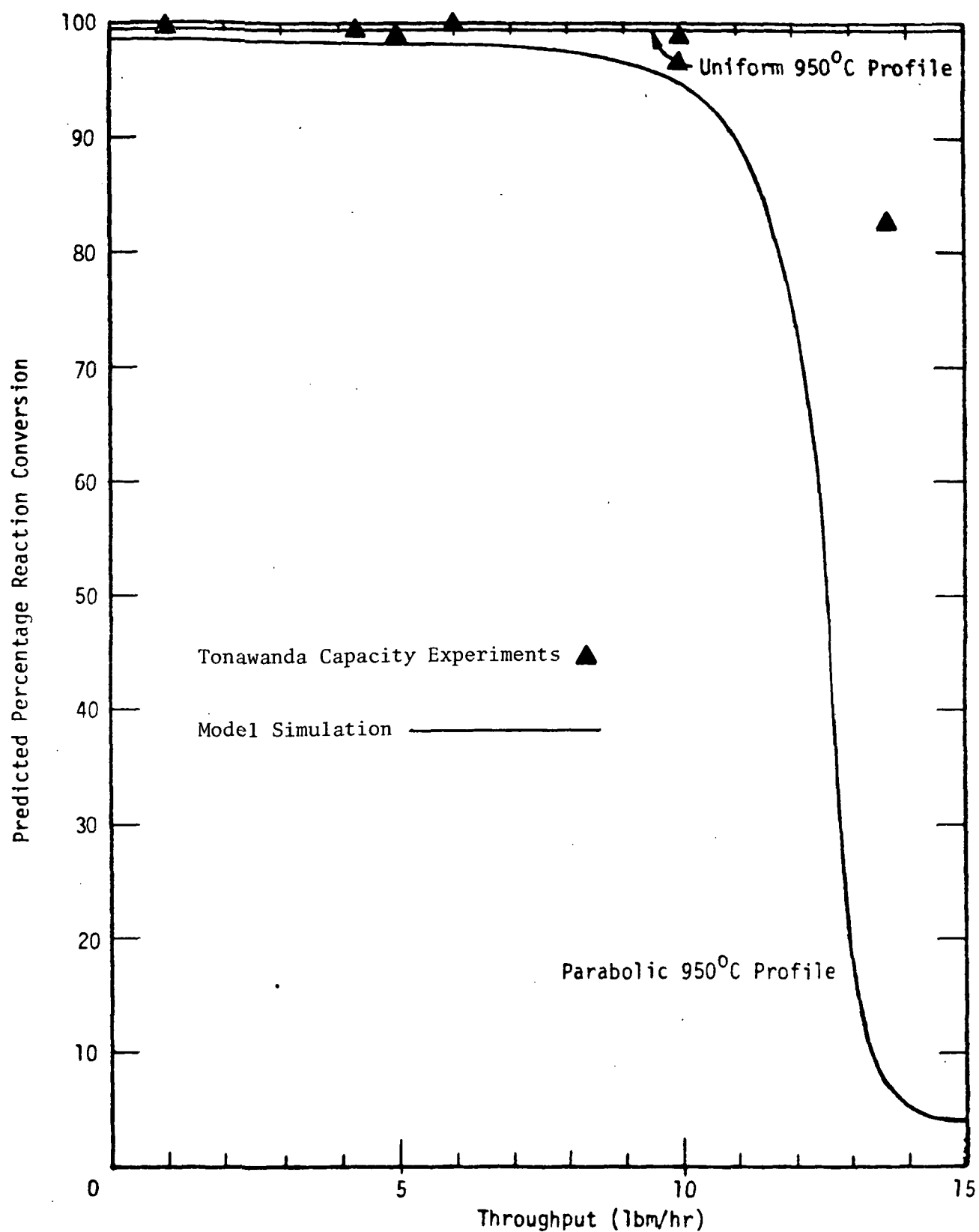
FIGURE 6.22   Effect of Wall Temperature Profile on Reactor Capacity

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with extensive experimental observations of wall buildup, has been used to define a criterion for reactor capacity based on the prevention of hard wall deposit formation.

The correlation relating the experimental observation of hard wall deposit formation to the maximum silane concentration near the reactor wall predicted by the model was established by the results shown in Figure 6.23. No hard wall deposit formation was observed experimentally in the PDU when the predicted silane concentration near the reactor wall was less than about 5%. The criterion for reactor scale-up is based on keeping the predicted silane concentrations near the reactor wall below the 5% level. The success of the Tonawanda experimental program in eliminating hard wall deposits stands as evidence of the validity of this criterion.

Since material constraints limit the maximum wall temperature that can be used, scale-up of the free-space reactor to EPSDU capacity must be accomplished by increasing the reactor size. Results from the theoretical model have been used to determine the minimum reactor diameter needed to successfully react the EPSDU throughput requirement. The following three criteria were used in scale-up of the reactor design:

- The temperature profile in the EPSDU reactor wall must be similar to that measured experimentally in the PDU.

- The flow field inside the EPSDU reactor must be similar to the flow field inside the PDU.

- The predicted silane concentrations near the reactor wall for the EPSDU design must be comparable or lower than levels predicted for PDU experiments where no hard wall buildup was observed (<5%).

These criteria were prescribed to ensure that the EPSDU reactor operate in a manner similar to the PDU, so that use could be made of the considerable operating experience with the smaller unit.
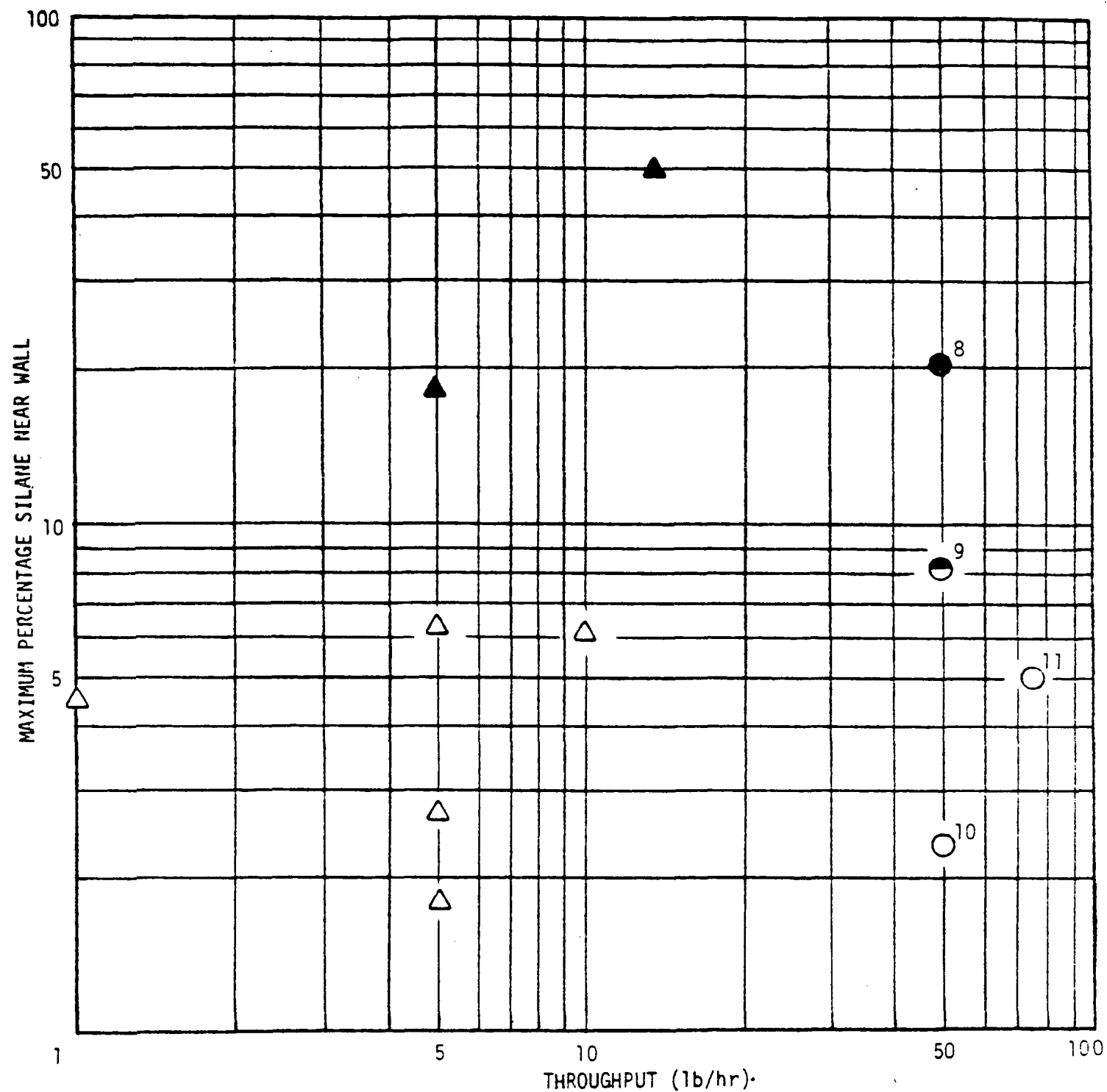
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FIGURE 6.23 Hard Wall Deposit Predictions for EPSDU Reactor Designs of
Various Diameters

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Predictions of wall buildup formation for the various reactor sizes are displayed in Figure 6.23. The corresponding points for several PDU experiments are included for reference. An 18" diameter reactor was the minimum size predicted to be capable of reacting 50 lbs/hr of silane on a continuous basis without hard wall deposit formation. The predicted maximum silane concentration near the wall of 2.3% is as low as any value predicted for any PDU experiment, so wall buildup should be minimal. Furthermore, an 18" reactor was predicted to be capable of successfully reacting 75 lbs/hr of silane with less than 5% silane near the wall, when the maximum wall temperature was raised to 925°C. On the basis of these results, an 18" inner diameter reactor was selected for the preliminary EPSDU design.

1.6.2 MELTING/CONSOLIDATION

The free-space reactor (FSR) produces a submicron-sized silicon powder by pyrolysis of silane gas. This product is impractical for use by silicon sheet manufacturers because of its low bulk density makes it difficult to store, package, transport and handle and is easy to contaminate. It is therefore mandatory that the powder be converted into a useful form by melting and consolidating into a pellet form.

1.6.2.1 Melting System Subcontract

Six candidate subcontractors were approached to respond to a request for proposal (RFP) which defined a statement of work and deliverables, etc. Two distinct objectives were required. The first consisted of an R&D effort required to experimentally demonstrate the operability of a melter system and the second was to prepare a functional design package for the EPSDU melter system. Technical proposals were received from four candidate subcontractors and were reviewed by an evaluation team which included a consultant. The proposals were rated for seven criteria, specifically: technical success, reliability and scale-up, product purity, 1986 cost goals, product form, fabrication facilities, and schedule.

The subcontract was awarded to Kayex Corporation, Rochester. The silicon consolidation scheme was based on melting the powder in a quartz crucible and dropping molten silicon shot from the crucible bottom into a cooling tower where the shot is solidified.

Subcontract work was started on March 1, 1980, and was completed in October, 1981. The goal of the project was to design, build, and test a melting/consolidation system suitable for installation in the EPSDU.

Initially, a small-scale set-up was used to prove the basic concept of melting silicon in chunk form by induction melting and dropping the melt through a nozzle in the base of a quartz crucible. After many tries and modifications, it was demonstrated that indeed chunk silicon and powder silicon could be melted and dropped through a nozzle. Based on this learning period, the experimental PDU system was designed and quotations for equipment were solicited.

The prototype system was fabricated, checked out, and the test program was started. Figure 6.24 shows a sketch of the general function of the melting/shotting system and Figure 6.25 shows a photograph of the system at Kayex.

The details of the equipment design, problems encountered, solutions to problems, description of testing and results have been amply documented by Kayex in their deliverable documentation. The contractual work is summarized in the following documents:

- Final Report

- Technology Development Report

- Functional Design Specification for SMS

- Operation Manual

- Design Package
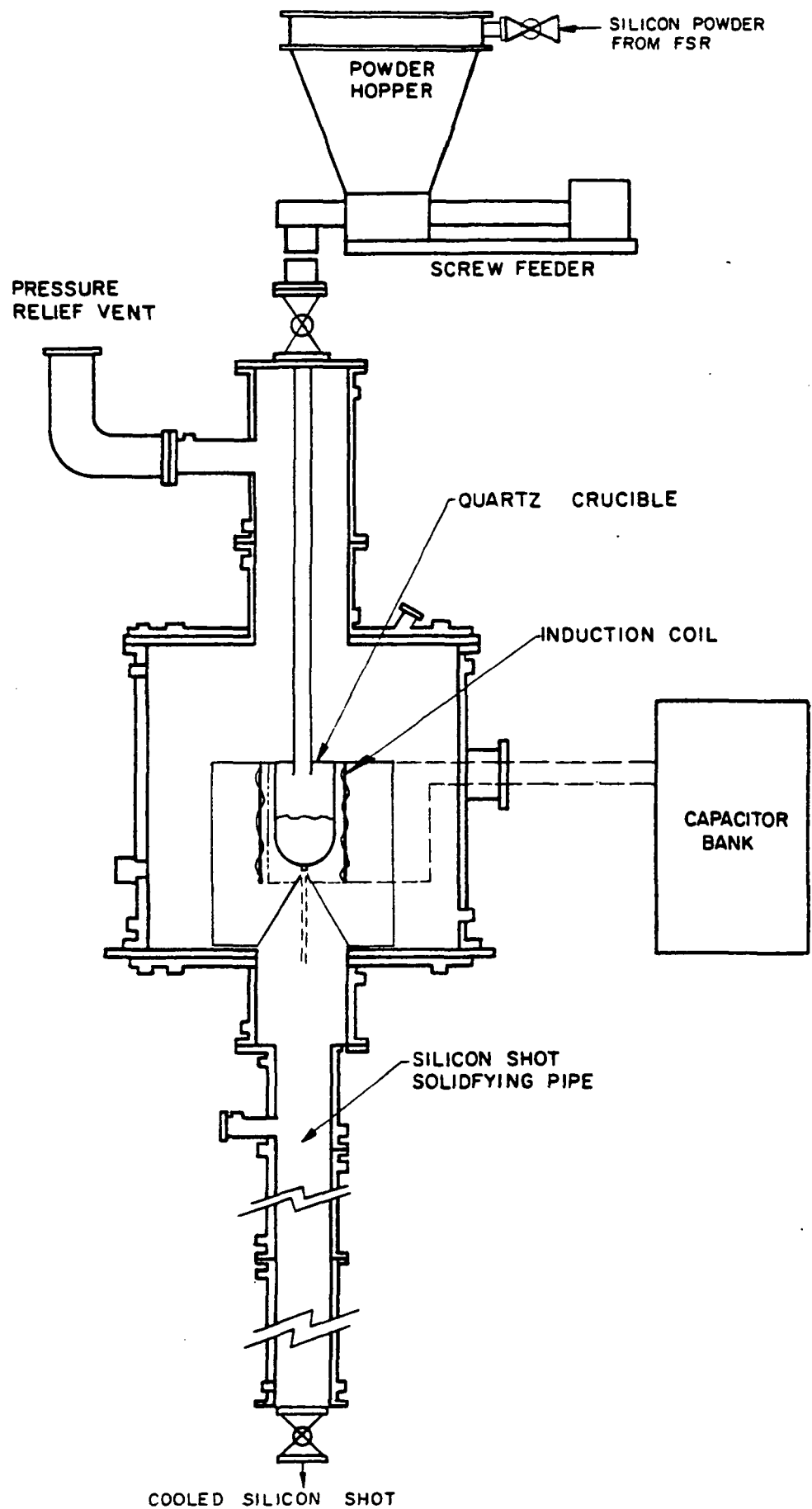
FIGURE 6.24   Silicon Powder Melting and Shotting System

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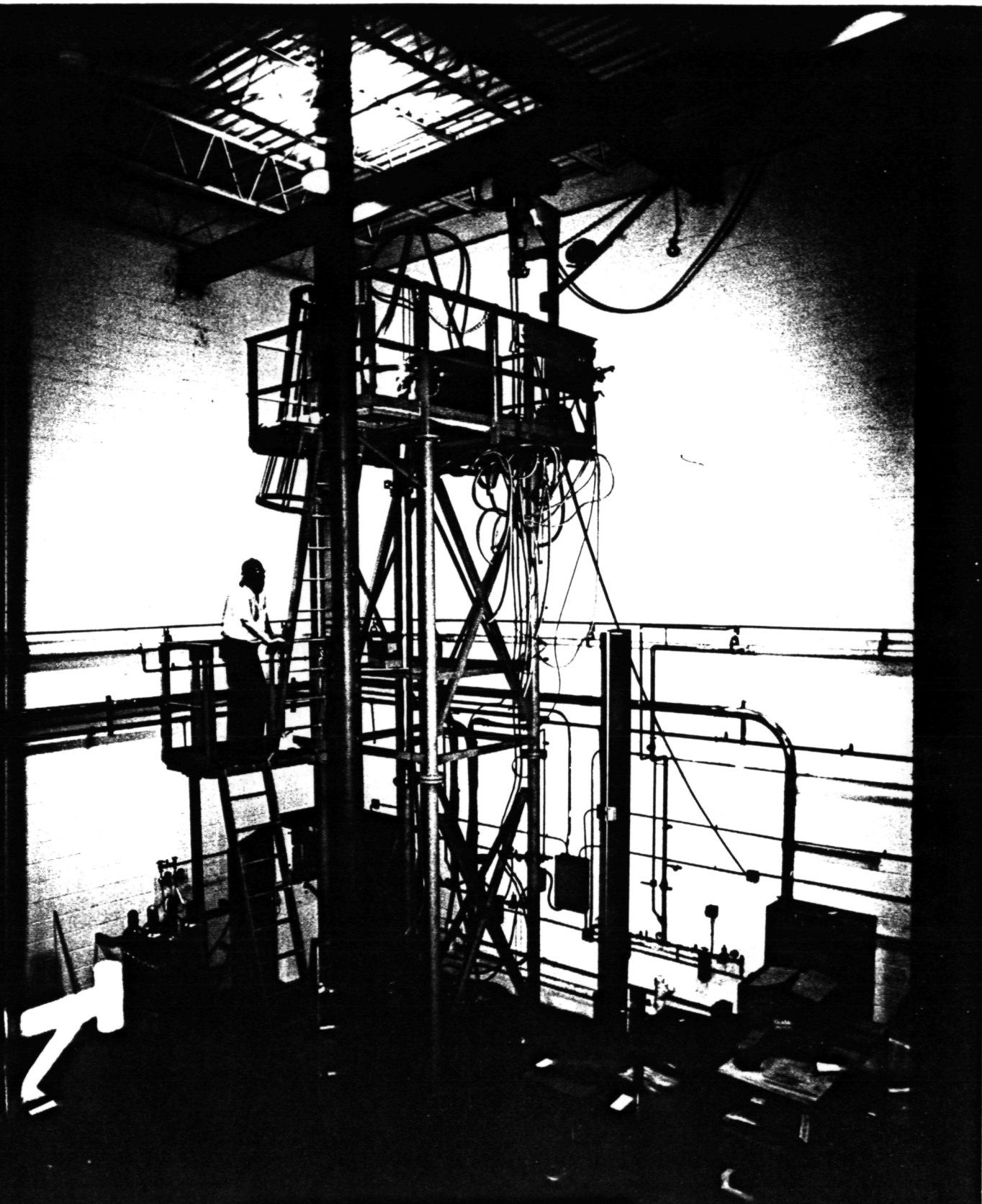
FIGURE 6.25 Melter/Shotter System

The following are comments on each of the documents:

- The final report by the Corporate Technology Center, Kayex Corporation, Rochester, New York, was submitted to Union Carbide.

- In addition, a Technology Development Report giving all details of testing and how adversities were overcome in the SMS development contract, was submitted to Union Carbide.

- .Functional Design Specification Package: This document was written to define a Silicon Melter System (SMS) suitable for consolidation of sub-micron sized silicon powder into "shot". The powder is a product of the free-space reactor (FSR) developed by UCC. The SMS was sized for 100 metric tons per year and the specification was based on the experience gained by Kayex in the design, fabrication and testing of a prototype SMS under sub-contract to UCC (Contract Number 825-50106).

  The prototype test results are, therefore, the basis and limiting factor in the selection and design for control methods, powder handling, "shot" collection, process parameters and throughput projections. It is evident that further study is required in projected design areas such as the use of multiple nozzles, auxiliary nozzle heaters and scale-up parameters, before confidence can be placed in a specification.

  The functional design specification package has the following contents:

  — General description and assumptions
      - overall operation assumptions
      - typical SMS sequence
      - major subsystems

— Design specification

   - general description

   - general specification

   - process streams - prototype system

— Utilities required

— Budgetary cost estimate

— Appendix - list of reference drawings and a drawing package

- **Operation Manual**: This document describes the operating procedures for startup, running and shutdown of the shotter/melter system.

- **Design Package**: The design package includes all detailed drawings for the shotter/melter system in its current configuration.

Summary

The program has proven that silicon powder as produced by the free-space reactor can be melted and consolidated in a shot form and that adequate purity for solar cells is practical. The experimental evidence to date still leaves some unresolved issues which require further development work prior to considering installing the PDU as an integral part of the EPSDU system, namely:

- melting powder and producing shot consistently in steady state

- throughput reliability and magnitude (kg/day)

- erosion rate of nozzles

- economics of operating multiple units (from 3 to 8) for an output of 100 MT/Yr

- demonstration of silicon shot purity over longer term operation

Due to lack of funding, further activity in this area was deferred.

1.6.2.2   Process Analysis (Melting/Consolidation)

UCC performed consolidation process analyses geared toward supporting the melter subcontract effort and EPSDU design activities. A theoretical analysis of silicon shot casting was conducted and the results of the analysis are summarized below:

The analytical work was concentrated in four major areas:

- Droplet formation
- Droplet cooling
- Process dynamics
- Cooling system requirements

Theoretical models were developed for each of these aspects of the shotting process. The size of droplets formed through an orifice was predicted for the region where drops form individually at the orifice, and for the varicose region where drops form by the breakup of a liquid jet (Figure 6.26). In the drop formation region, EPSDU throughput of 16 kg/hr can be met with a single orifice (diameter between 1.5 and 2.0 mm) forming droplets approximately 5 mm in diameter as can be seen in Figure 6.27. In the varicose region, the throughput requirement of EPSDU can be satisfied by a single orifice with a diameter of 0.5 to 1.0 mm that gives rise to droplets 1 to 2 mm in diameter. Table XI shows the upper and lower bounds on the varicose region for different orifice diameters, and also lists the predicted droplet sizes.

The solidification of droplets was modeled to determine the lower height required for complete solidification. The model predicts the temperature profile in the solidifying droplet, the velocity and position of the droplet, and the solidified shell thickness of functions of time. Figure 6.28 shows the tower height required for 100% droplet solidification.

The tower height required increases exponentially as the droplet diameter increases. The high thermal conductivities of hydrogen and
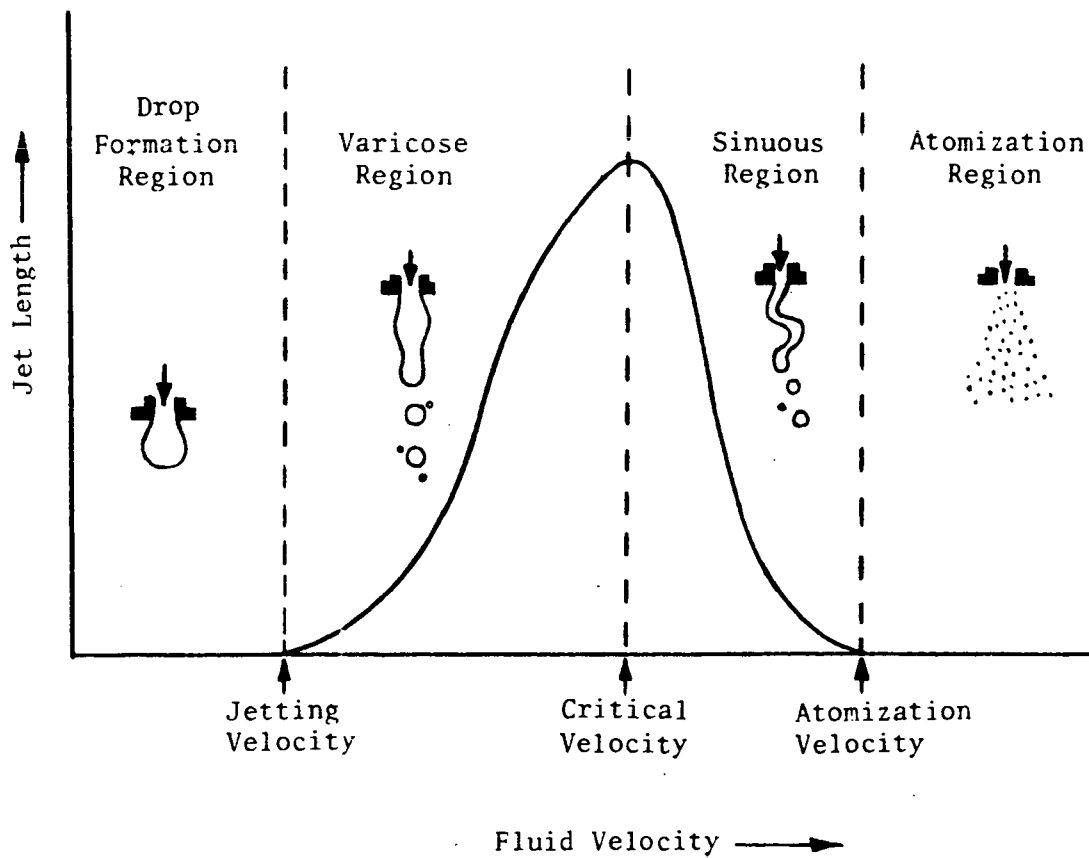
FIGURE 6.26   Jet Length Vs. Velocity for Fluid Flow Through an Orifice
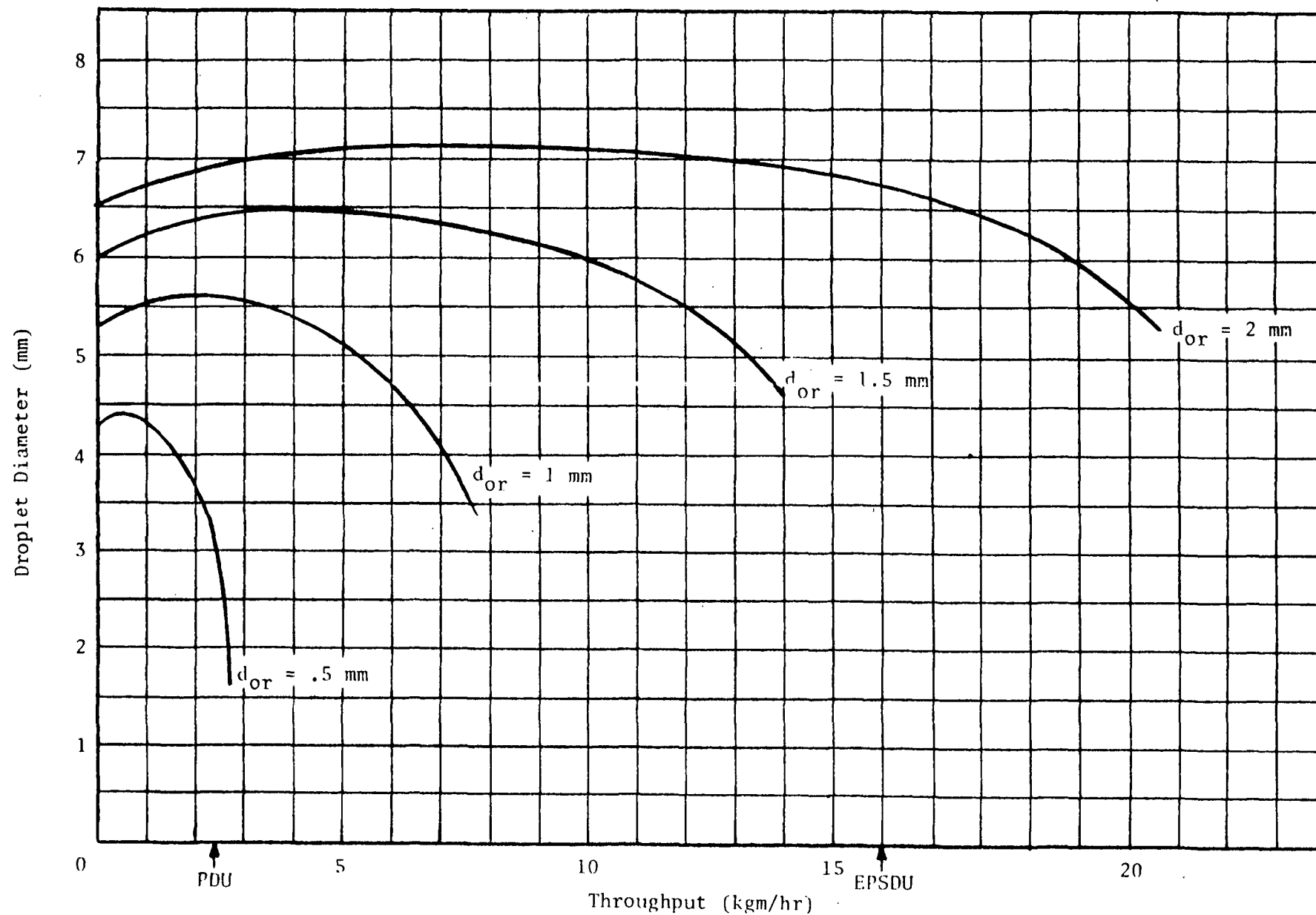
FIGURE 6.27 Prediction of Size of Droplet Formed in Drop Formation Region

TABLE XI

DROPLET SIZES IN THE VARICOSE REGION
(by analysis of model)

Orifice Diameter (mm)	Throughput at Jetting Velocity (kgm/hr)	Throughput at Atomization Velocity (kgm/hr)	Predicted Droplet Size (mm)
0.5	2.7	104.6	0.95
1.0	7.6	276.7	1.89
1.5	14.0	562.0	2.84
2.0	21.6	738.3	3.78

(EPSDU throughput: 15.9 kgm/hr)
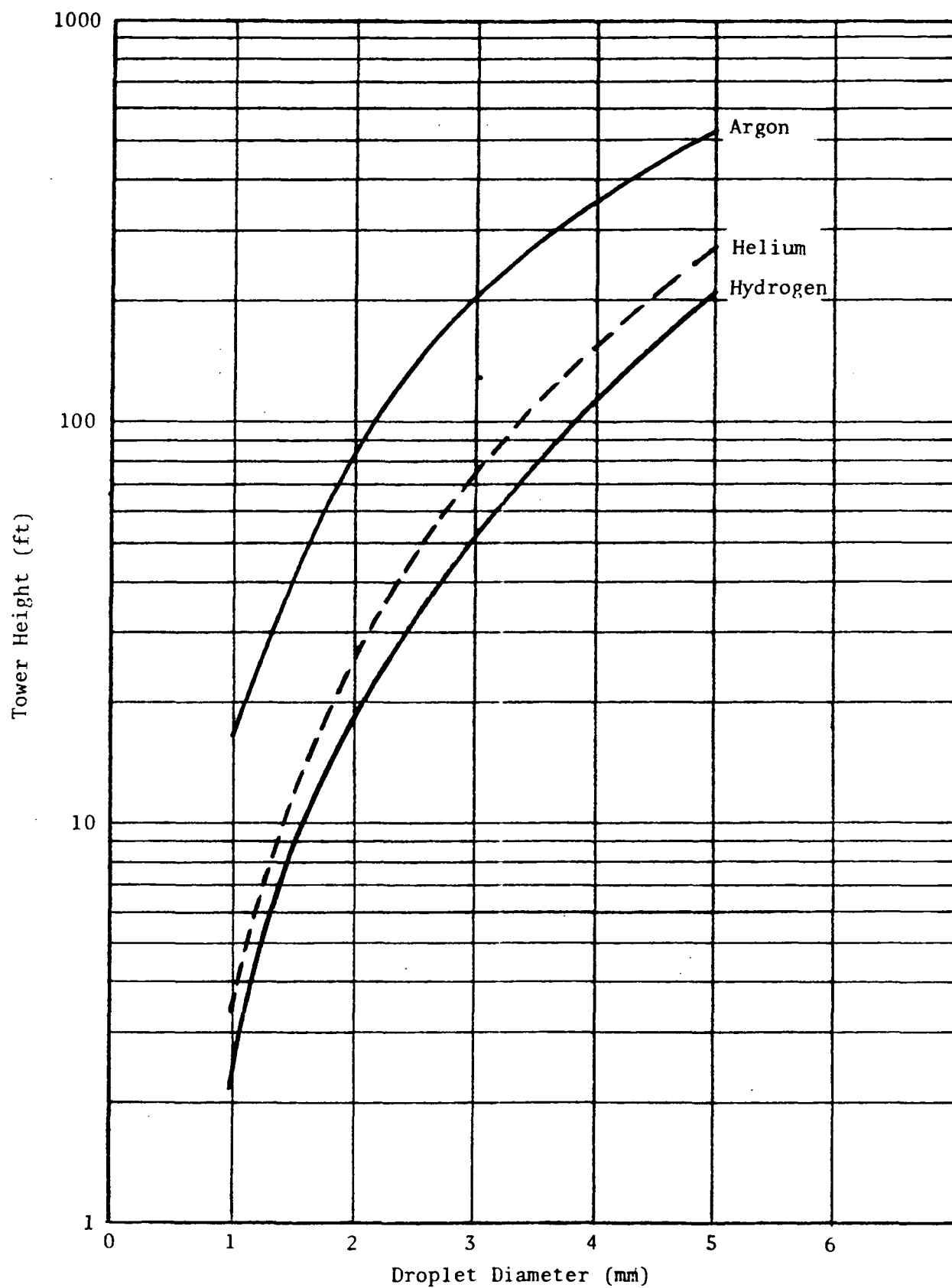
FIGURE 6.28   Tower Height Required for 100% Solidification

helium dramatically increase the amount of convective heat transfer from the droplet over that observed in argon, and result in more rapid cooling of the droplet. Consequently, despite the higher terminal velocities of the droplet, the tower height required for complete solidification of a given size drop is less if hydrogen or helium is used as the cooling gas.

The results clearly show that if the tower height is to be kept under 20 feet, the droplets formed must be less than 2 mm in diameter. Droplets produced through a 1 mm diameter orifice at EPSDU throughputs in the drop formation region are approximately 5 mm in diameter. Figure 6.28 shows that a tower of at least 200 feet in height is required to completely solidify a 5 mm droplet.

Figure 6.29 shows the temperature profile in a 2 mm droplet solidifying in hydrogen. The phase front moves inward more rapidly as solidification progresses since less mass is contained in spherical shells close to the center of the droplet than in those shells near the outer radius.

A steep temperature gradient exists in the droplet during solidification which may lead to severe thermal stresses in the solidified shell. In addition, since liquid silicon expands as it solidifies, additive tensile stresses are present. The total induced stress may be large enough to fracture the droplet. Cracking of the solidified shell is not perceived to be a serious problem, provided the droplet does not shatter into small fragments. A detailed analysis of thermal stresses in the droplet has not been performed, but papers dealing with the subject have appeared in the literature. It is clear from the droplet cooling analysis that if droplets of the desired size range (greater than 1 mm) are to be completely solidified in a 20-foot high tower, then cooling must be achieved at a maximum possible rate. If shattering of the droplets proves to be a serious problem, then the thermal stress problem should be investigated further.

The dynamic response of the shotting system to changes in the powder feed rate, pressure differential across the melt, and orifice diameter was determined. The melt height has a very slow response to changes in the orifice cross-sectional area, which indicates that plugging or erosion of the nozzle cannot be adequately monitored by monitoring the melt height. Monitoring of the droplet throughput and size is suggested as the best way of determining the orifice condition.
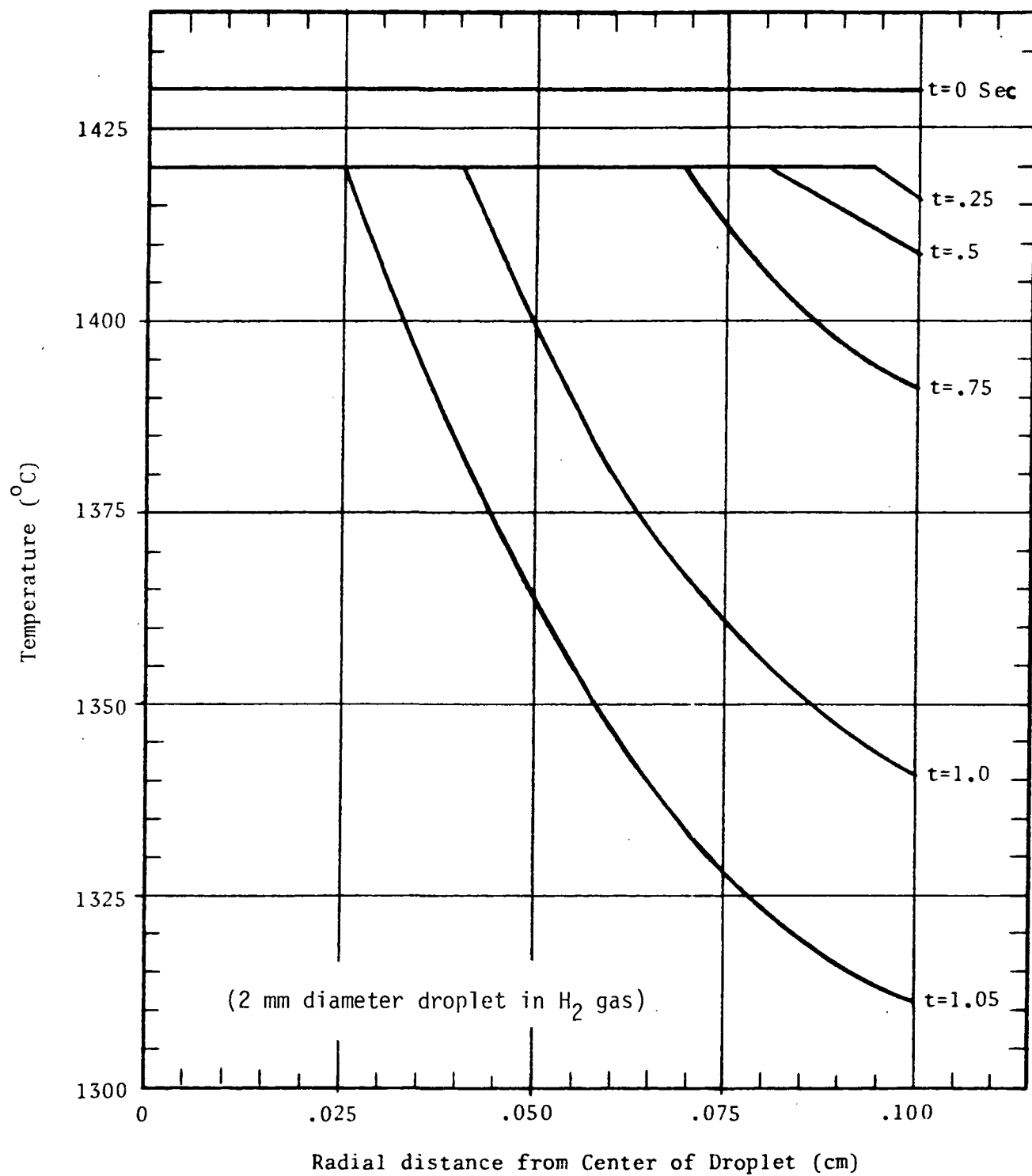
FIGURE 6.29   Temperature Profiles in Solidifying Droplet

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The cooling requirements for the shotting tower were predicted to establish design parameters. The cooling requirements were shown to be minimal provided the melting furnace is properly insulated from the tower.

System Compatibility Requirements for a Hydrogen Atmosphere

A study of the melter compatibility for hydrogen atmosphere operation was conducted. This study included a recommendation on the electrical classification for the melter building assuming an all-hydrogen environment inside the melter. A second part of the study examined the compatibility of melter materials for operation in a hydrogen atmosphere.

The following features were noted for the EPSDU melter area:

- Produce purity requirements necessitate the tightest possible system

- System pressures are low (10 psi or less)

- Most process lines are small

- Mechanical ventilation of at least 6 air changes per hour will be supplied

In light of these considerations, the low-bay portion of the building could be electrically unclassified. If a hazardous accumulation of hydrogen were to occur, it would be in the upper level of the high-bay. Therefore, it was recommended that the high-bay area be handled as a Division 2 which requires the elimination or proper enclosure of sparking devices and the use of vapor-tight lighting fixtures.

The capacitor bank for the induction power supply and screw feeder/motor assembly should now meet Class 1, Division 2, Group B classification. The control panel for housing instruments and controls need not meet Division 2 requirements since it will be located in an electrically unclassified area at EPSDU. However, since the atmosphere around the panel could get dusty, the instrumentation and controls should be housed in a dustproof box such as a Hoffman free-standing NEMA Type 12 enclosure.

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Summary

- Shot larger than 2 mm in diameter cannot be completely solidified in a 20-foot high cooling tower.

- Shot of the desired size (1 - 2 mm diameter) can be formed at rates comparable to the proposed EPSDU throughput (15.9 kgm/hr) by forcing molten silicon through a 1 mm orifice at velocities corresponding to the varicose region, where drops form by jet breakup.

- Droplets formed at low flow velocities through orifices of reasonable size are too large to solidfy completely.

- Hydrogen or helium was selected as the desired tower cooling gas because of enhanced heat transfer characteristics.

- The results obtained from the theoretical model were used by Kayex to guide the design of the shotting system.

1.6.3   FLUID BED DEVELOPMENT

This development program includes all analytical, experimental, and design effort associated with developing a fluid-bed reactor as an alternative or backup system to the Free-Space Reactor.

The R&D program for the fluid bed development consisted of three parts:

- A small fixed-bed deposition study to obtain silane reaction rate data, the critical silane concentration curve and silicon plating morphology.

- An inert gas fluidization study to test arc heating techniques and determine particle separation boot parameters.

- A 6-inch diameter fluid bed PDU to acquire optimum
  operating conditions, prove continuous operation,
  test for product purity and gather scaling parameters
  necessary for designing a commercial-size fluid bed
  pyrolysis system.

I.6.3.1   Fixed-Bed Deposition

Introduction

The purpose of the fixed-bed experiments was to obtain data re-
quired for the design and evaluation of the fluid bed process for silane
pyrolysis. The data was used to design a first-generation fluid-bed silane
pyrolysis development unit.

Although fixed-bed experiments do provide meaningful data that
can be obtained with modest expenditure, there are some disadvantages. It is
difficult to maintain constant and uniform temperatures in a fixed bed because
the bed is heated externally and the reactor wall temperatures are higher than
the bed temperatures. However, by careful design and operating procedures,
these bad effects were minimized and the experiments were successfuly completed.

The specific objectives of the fixed-bed experiments were:

- to determine heterogeneous deposition rate of silicon,

- to define bed temperatures and silane feed concentra-
  tions which favor heterogeneous decomposition and
  minimize the homogeneous mode,

- to qualitatively describe deposition morphology.

The results of this silane decomposition study generated a critical silane concentration curve, which is defined as the boundary between homogeneous and heterogeneous decomposition. The critical silane concentration curve, a qualitative study of the morphology of the decomposition aided by the use of a scanning electron microscope, and deposition rate measurements are described herein.

Summary

The decomposition rate measurements agreed favorably with the literature, homogeneous decomposition was minimized by determing the dependency of decomposition on silane concentration and temperature, and dense, coherent deposition was obtained.

The deposition rate measurements showed the decomposition of silane to be a first-order irreversible reaction within the range of experimental conditions. The rate measurements also show no composition or velocity dependence in the reaction in the range of data collected. The results of the kinetic rate study compared well with literature, especially with the work of Eversteijn.[1] The reaction rate constantly varied from 0.010 (min^{-1}) at 576°C to 0.112 (min^{-1}) at 676°C. Also, a further comparison shows the activation energy for heterogeneous decomposition to be 39 kcal/mole from this kinetic rate study (versus 37 from Eversteijn's work).

The dependency of silane homogeneous or heterogeneous decomposition on the concentration of silane and the temperature is described as the critical silane concentration curve (Figure 6.30). The distinction between homogeneous and heterogeneous decomposition, which is presented as a band instead of a definitive line because of the selection of the temperatures, seems to be an extension of data obtained by Murthy, et al.[2] At 20% silane the temperature band is 650 to 670°C. At 10% silane the temperature band is 700 to 750°C, and at 1% silane the temperature band is 820 to 895°C. These results were determined with a packed bed of high-purity unetched silicon

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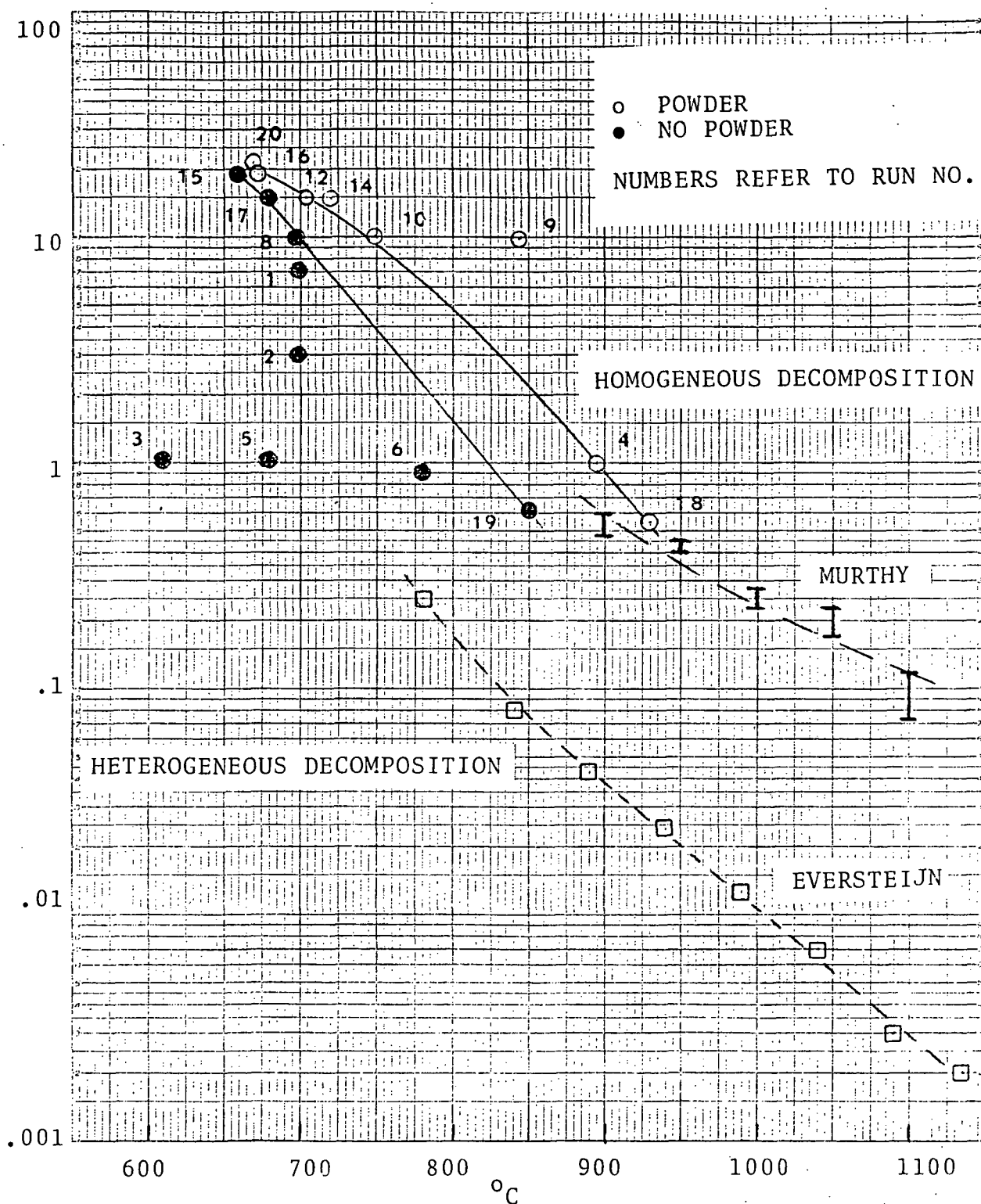
FIGURE 6.30 Temperature Dependence of Critical
Silane Concentration for Packed Bed
of High-Purity Unetched Silicon

particles; however, the use of acid-washed and etched silicon particles gave somewhat different results. The heterogeneous decomposition region was lowered by approximately 50°C (Figure 6.31). The critical silane concentration curve for etched high-purity silicon lies somewhere between the data of Murthy et al[2] and Eversteijn[3].

Heterogeneous decomposition using unetched silicon particles in the bed resulted in nodular and fiber silicon growth with the dominant type being fibrous (Figure 6.32). Use of acid-washed and etched silicon particles in the bed, strongly reduced fiber growth (Figure 6.33). The silicon deposits on the etched particles were dense, coherent, and well attached to the particle surface. Analysis of the acid-washed and etched silicon particle surface indicated one order magnitude reduction of metal contamination from 50 ppm to 5 ppm) and removal of surface residue (Figure 6.34). These factors must be contributing to the favorable reduction of surface fiber growth.

Experimental Results

A. Critical Silane Concentration Curve Determination

The critical silane concentration curve is a function of both feed composition and temperature, and distinguishes whether decomposition is homogeneous or heterogeneous. Heterogeneous decomposition is the deposition of silicon onto the packed bed silicon particles, while the result of homogeneous decomposition is the formation of sub-micron silicon powder.

There are two factors to consider in the collection of data for the critical silane concentration curve. First, the distinction between homogeneous and heterogeneous decomposition is presented as a band instead of a definitive line. This is because each run is made at a constant temperature and silane concentration. The result of each run is then interpreted individually as homogeneous or heterogeneous decomposition. The temperature is continually adjusted in successive runs until a narrow band between
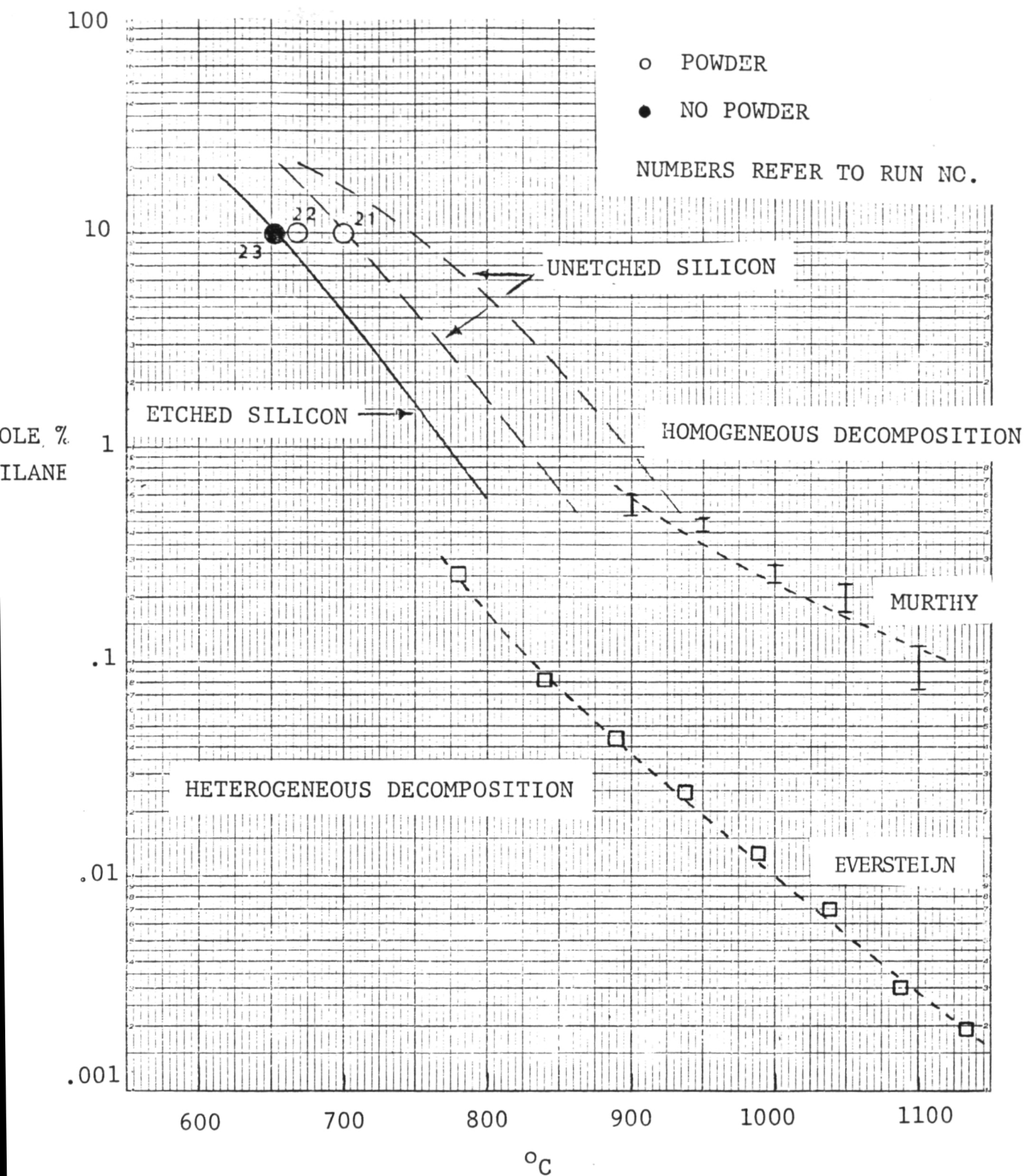
FIGURE 6.31 Temperature Dependence of Critical Silane
Concentration for Packed Bed of Etched
High-Purity Silicon
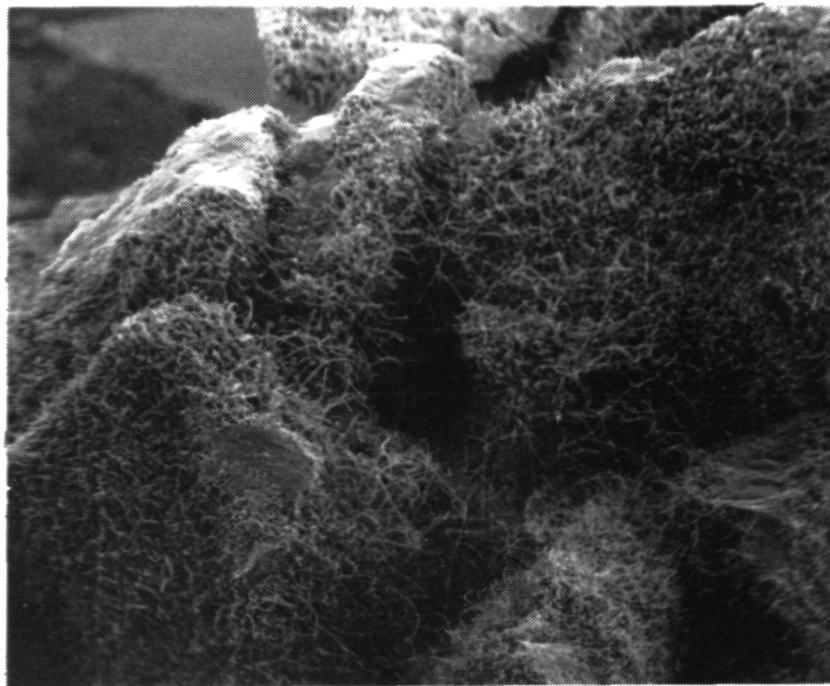
30X
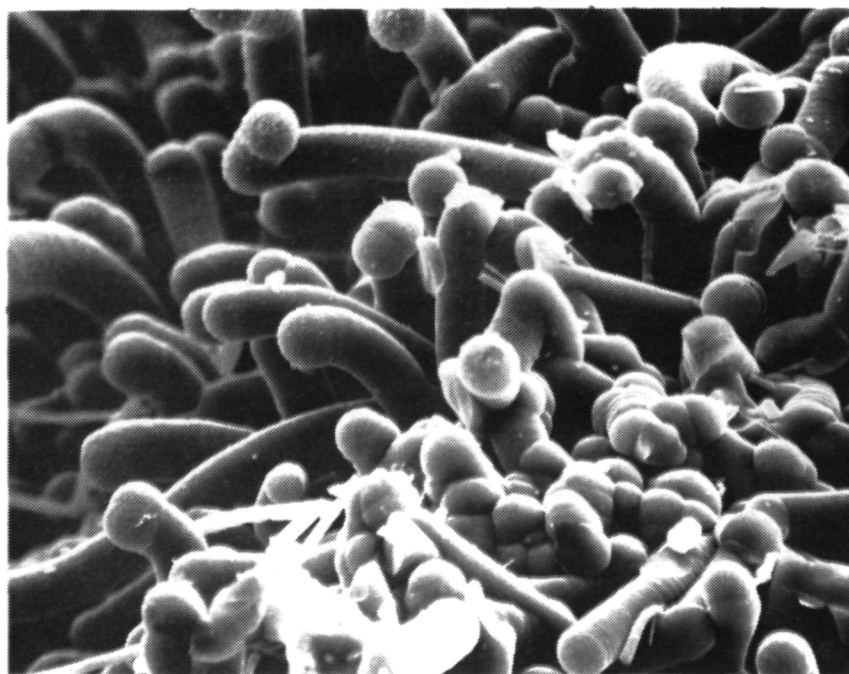
700X

FIGURE 6.32   Scanning Electron Photomicrograph of High-Purity
Unetched Silicon Particle Surface after Heterogeneous
Silane Decomposition.   Run 8, 9.8% Silane, 696°C.
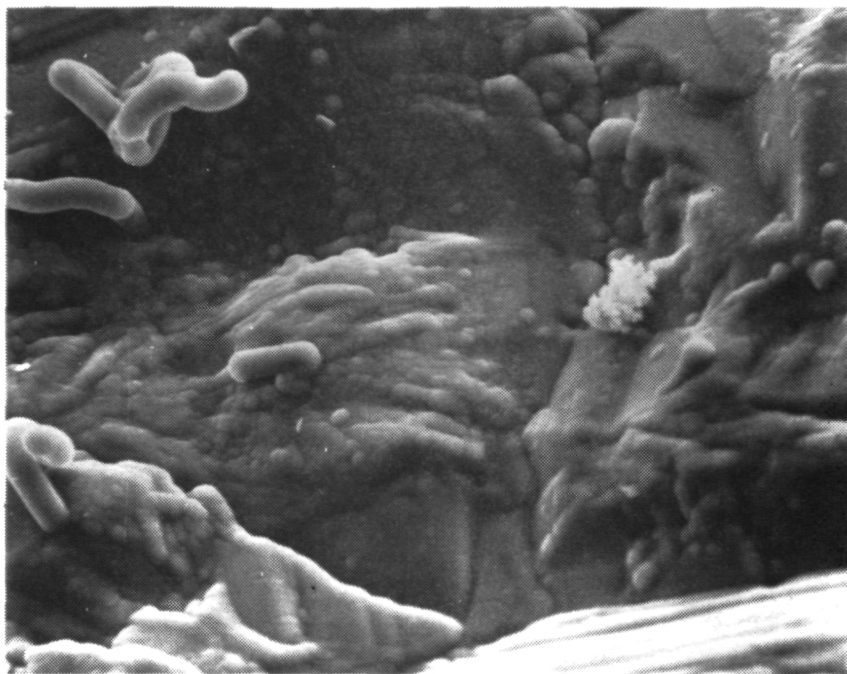
500X
Run 23
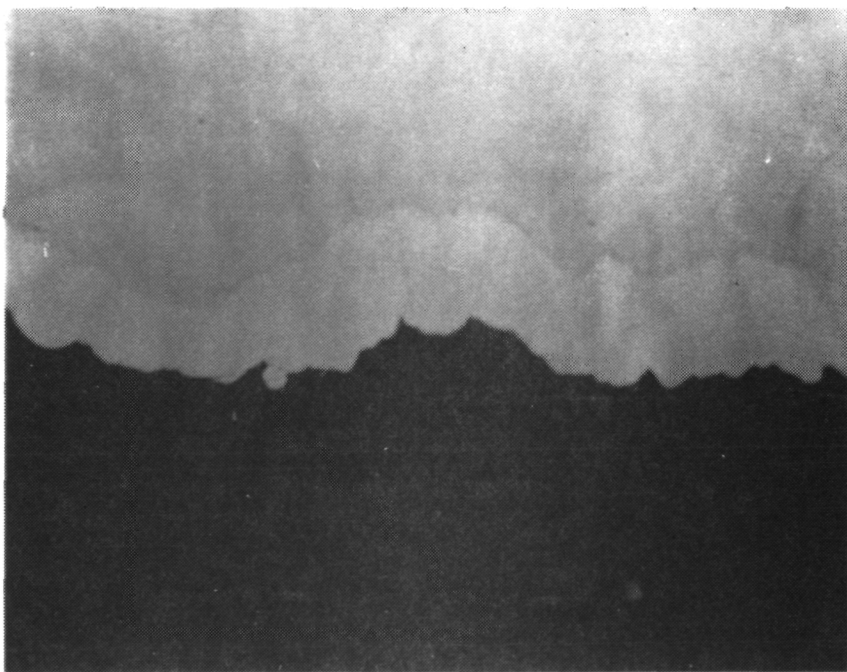
500X
Run 23

FIGURE 6.33   Scanning Electron Photomicrograph of Cross-Sectioned
Etched High-Purity Silicon Particle Surface after
Heterogeneous Silane Decomposition. Run 23, 9.8%
Silane & 652°C.
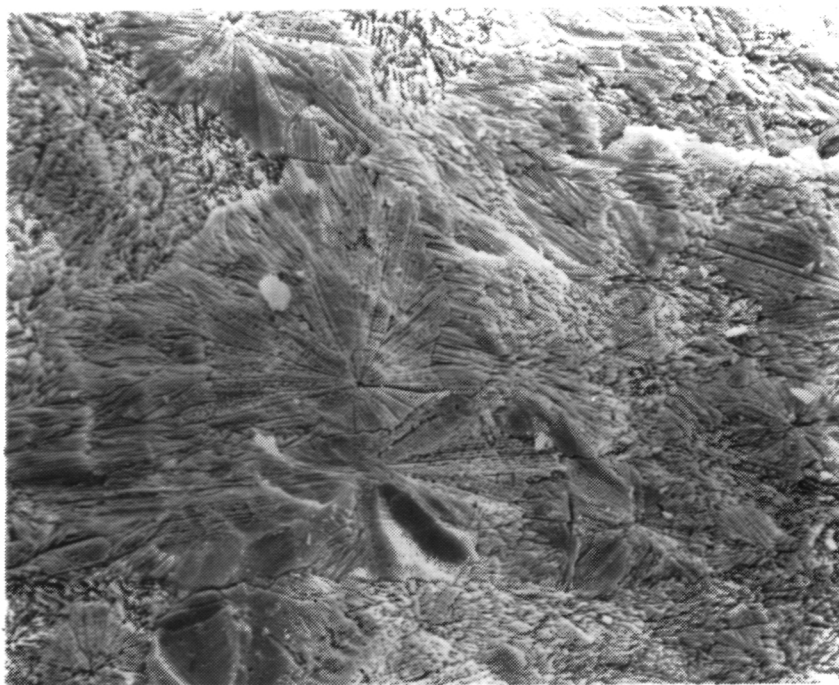
500X
ETCHED
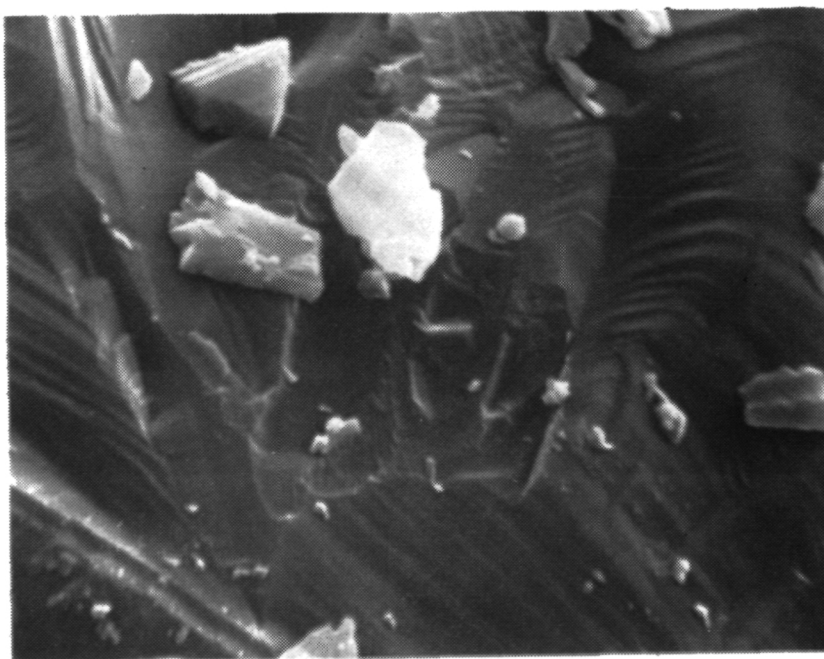
700X

UNETCHED

FIGURE 6.34   Scanning Electron Micrograph of Etched & Unetched
High-Purity Silicon Particle Surface Before Silicon
Deposition

homogeneous and heterogeneous decomposition is determined for a given silane concentration. Second, most of the data was taken initially with a packed bed of high-purity unetched silicon. It was realized later that cleaned and etched high-purity silicon would give different results and affect the operation of a fluidized bed silane pyrolysis unit.

B. Critical Silane Concentration Curve Results - Unetched Silicon

Figure 6.30 shows the resulting correlation from the experimental data between temperature and the composition of silane. The two lines connecting the solid and open circles denotes the band separating the homogeneous from the heterogeneous decomposition region. The critical silane concentration curve lies somewhere in this band between 1.5 to 5% silane at $800^{\circ}C$, 10 to 16% at $700^{\circ}C$ and much above 20% at $600^{\circ}C$.

The criterion used to determine homogeneous decomposition is the appearance of powder in the packed bed after the reaction is finished. This was accomplished by removing the packed bed from the quartz liner and examining the particles either visually or by a scanning electron micrograph. In the majority of cases, powder formation was also confirmed by a rapid rise in bed ΔP.

Also shown on Figure 6.30 is the work of Murthy and Eversteijn. They determined the critical silane concentration curve by slowly increasing silane concentration in a constant temperature reactor until powder was visually observed. Figure 6.30 shows that the data from this work is an extension of Murthy's data at temperatures below $900^{\circ}C$.

One assumption was made in the interpretation of the results of this work; mainly, that the amount of silane depleted from the bed stream is negligible before the feed stream is heated to bed temperature. This is not true because some silicon is deposited on the inner quartz-liner wall about ¼-inch above the top of the packed bed. Also, because of heat transfer limitations, the feed stream must pass through a few layers of packed-bed particles before gas temperature approaches bed temperature.

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Since the surface area of the quartz liner wall where the silicon deposited is small compared to the surface area of the bed particles, silane loss was considered negligible. Also, since silane passed through only a few layers of packed bed before reaching the bed temperature, it was assumed to enter at the bed temperature.

C. Deposition Morphology - Unetched Silicon

The scanning electron micrographs of high-purity unetched silicon particle surfaces, after the silane reaction, shows the main form of heterogeneous decomposition to be fiber growth. Figure 6.32 is a typical illustration. Fiber growth was also noted in previous packed-bed work at Parma[4] and by other investigators[5]. Some nodular growth and plating was also seen and is the preferred type of morphology; however, the dominant growth is by fibers.

In homogeneous decomposition, brown powder is the end result of the silane reaction but at times fiber growth also accompanies the powder as seen in Figure 6.35. As the temperature increases at a constant silane composition, the fibers become smaller in diameter. If the temperature is well into the homogeneous region, only powder remains as shown in Figure 6.36; no fibers are formed.

D. Critical Silane Concentration Curve Results - Etched Silicon

After many tests with high-purity silicon, it was apparent that fiber formation was the dominant heterogeneous growth resulting from silane decomposition. A Japanese work[5] implied that metal impurities and other surface residue provided nucleation points for fiber growth, and if the surfaces are clean, fibers are eliminated. Since our silicon particles were prepared by grinding between two serrated steel plates, there was iron contamination on the silicon surfaces. To see what effect clean surfaces would have on the critical silane concentration curve data, a sample of high-purity silicon was acid-washed and etched. Figure 6.31 shows the experimental results of using high-purity silicon that has been acid-washed and etched. The lower edge of the temperature band of the critical silane concentration curve at 9.8 mole % silane was lowered approximately 50°C.
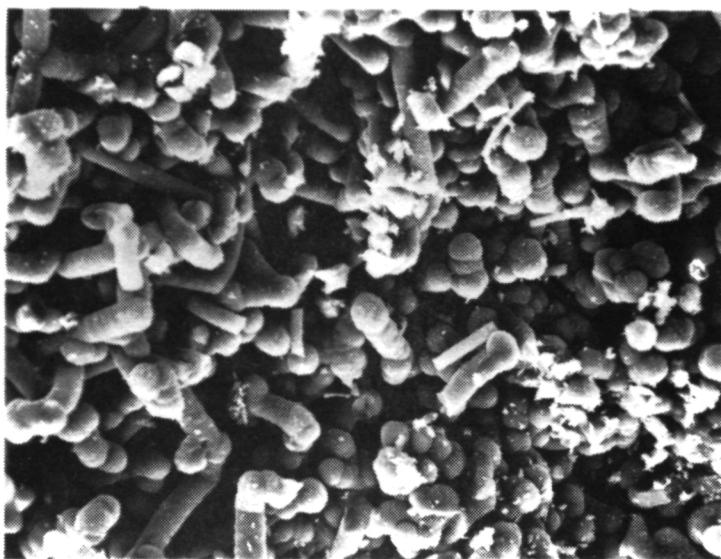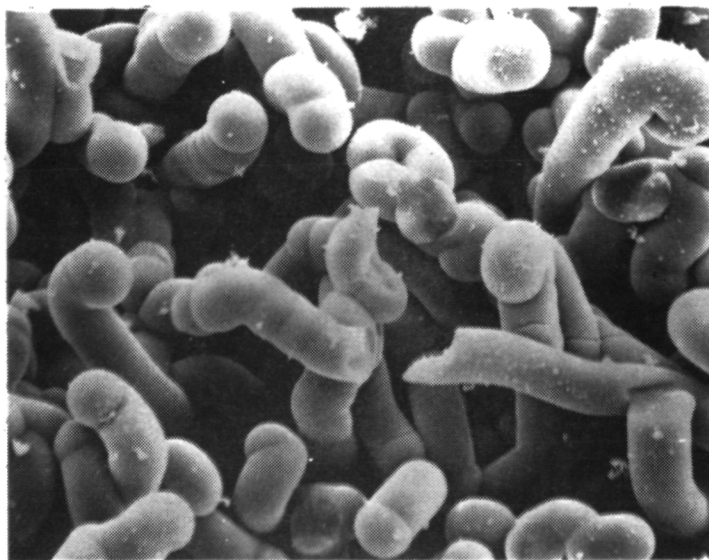
500X
Run 14

500X
Run 12

FIGURE 6.35   Scanning Electron Micrograph of High-Purity Unetched
Silicon Particle Surface after Homogeneous Silane
Decomposition.   Run 12, 15.0% Silane and 704°C.
Run 14, 14.8% Silane and 720°C.
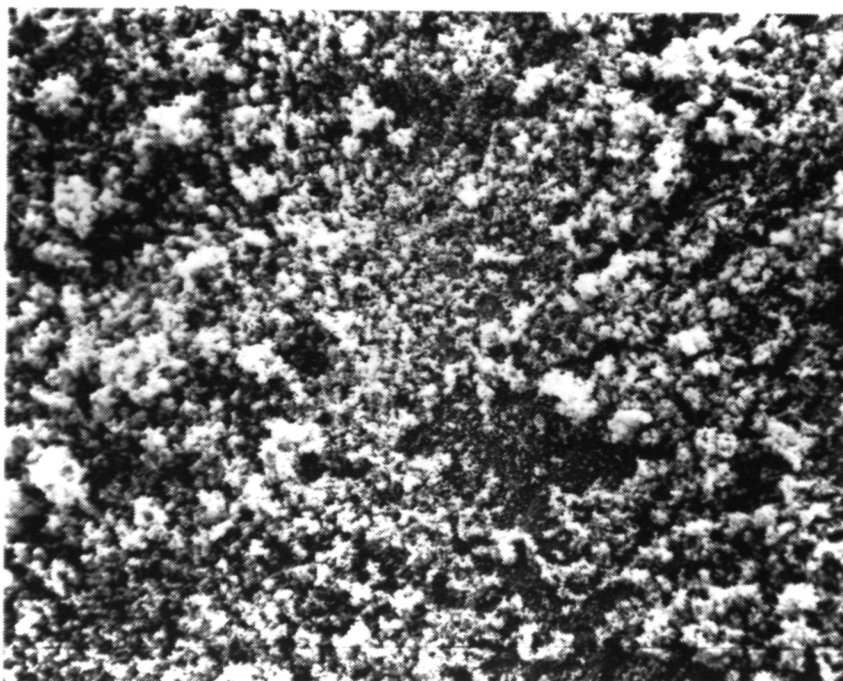
500X
Run 18

FIGURE 6.36   Scanning Electron Micrograph of Homogeneous Silane
Decomposition.   Run 18, 0.55% Silane and 930$^{\mathrm{o}}$C.

Experiments were conducted only at 9.8 mole % silane with the band between homogeneous and heterogeneous silane decomposition estimated by following the same slope as the experimental results using unetched high-purity silicon. Now, the data from this work falls between the results of Murthy and Eversteijn.

E.   Silicon Deposition Morphology - Etched Silicon

A result of acid-washing and etching the bed of silicon particles was the dramatic reduction of fiber growth. A comparison of Figure 6.33, etched silicon, with Figure 6.32, unetched silicon, shows the vast reduction in fiber growth on the silicon surface. Also shown in Figure 6.33 is the polished cross-section of a silicon particle. The silicon deposition is dense, firmly attached to the silicon particle surface and averages about 25 microns in thickness. This is the type of silicon plating which is desired in the fluid-bed pyrolysis unit.

Figure 6.37 illustrates the result of homogeneous decomposition. The scanning electron micrograph reveals balls or clusters of powder material lying on the particle surface. The photomicrograph shows these clusters to be loosely attached to the etched silicon surface.

The appearance of the silicon particle surface before silane decomposition for etched and unetched high-purity silicon is shown in Figure 6.34. The unetched particle surface has clear and distinct cleavage lines and shows silicon particle debris resulting from the grinding operation. After etching, the debris is eliminated along with the cleavage lines.

F.   Silane Heterogeneous Decomposition Reaction Rate

Three conditions must be satisfied before reliable reaction rate data can be taken with the present apparatus. First, the temperature distribution throughout the length of the bed must be uniform; the maximum temperature spread in the current experiments was controlled to within 10°C or less. Second, the deviation of average bed temperature for a series of tests at some pre-determined constant temperature must be kept small; this amounted to $\pm 3^{\circ}$C in the rate data presented in this report.

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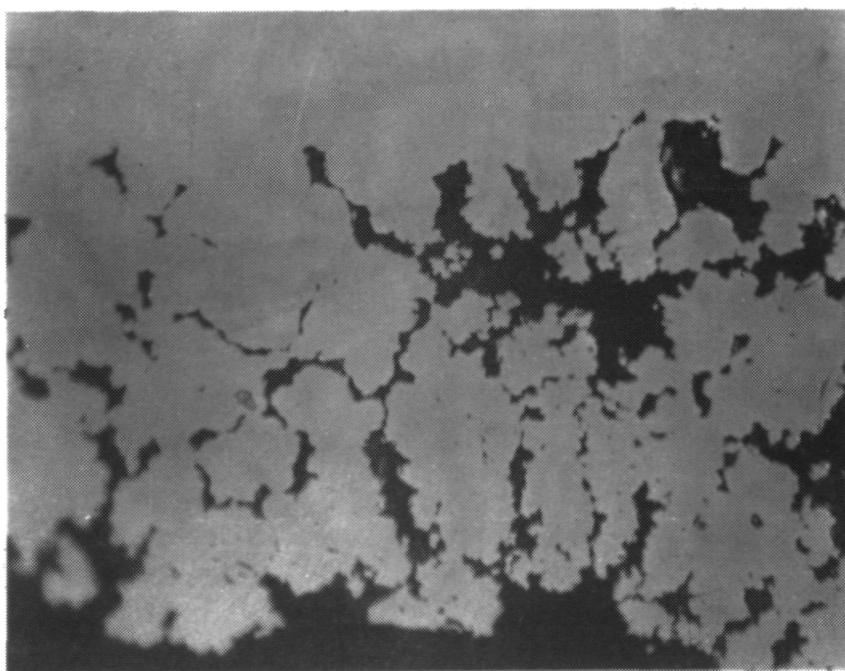
500X
Run 21

500X
Run 21

FIGURE 6.37   Scanning Electron Micrograph & Photomicrograph of
Cross-Sectioned Etched High-Purity Silicon Particle
Surface After Homogeneous Silane Decomposition.
Run 21, 9.8% Silane and 701°C.

Third, the hot effluent silane-hydrogen gas mixture from the fixed bed must be quickly quenched to below 400°C or else the silane will continue to decompose and a true silane composition analysis cannot be made.

Silane decomposition rates are measured by noting the reduction in silane concentration as the gas stream passes through a packed bed of silicon particles, all at some pre-determined constant temperature and flow velocity. Bed temperature is held constant and the flow velocity changed. The change in outlet silane concentration is again noted. This procedure is performed for several runs. The next course of action is the search for a rate equation. Two conditions must be satisfied: (1) is the reaction rate model correct and (2) what are the best values of the rate constant. These questions are best answered when the rate equations are put into a linear form. If calculated data points deviate from a straight line, then the proposed rate model is incorrect and another model is tried. Fortunately, the rate equations for a plug flow reactor are easily integrated and linear equation is obtained. The rate constant is then found by determining the best slope of the straight line utilizing a least square regression of the deviation of the data points from the straight line.

Conclusions and Recommendations

The fixed-bed experiments have supplied valuable information for the design of the forthcoming first-generation fluid-bed silane pyrolysis development unit. Important information was gathered in the areas of silane decomposition rates, heterogeneous versus homogeneous silane decomposition, and the morphology of heterogeneous decomposition.

The fixed-bed was initially difficult to operate properly. However, after stable temperature equilibrium was established throughout the bed and other operational techniques improved, reliable data was obtained. Acid washing and etching of the high-purity silicon particles definitely affected the results of the critical silane concentration curve (heterogeneous versus homogeneous decomposition) and probably the rate data.

All further fluid-bed work should be performed with high-purity silicon which has been acid washed and etched after the silicon is ground and screened.

1.6.3.2   Inert Gas Fluidization

The experimental program, conducted in support of a fluid-bed silane pyrolysis PDU, was successfully completed; it demonstrated that a fluidized bed has potential for the production of silicon. A description of the experiments and results are presented.

1.6.3.2.1   Capacitive Heating Tests

<u>Introduction</u>

Capacitive electrical heating is an important feature of the fluid-bed reactor for silane pyrolysis. By directly heating the bed, a particle temperature higher than the reactor wall temperature may be established which should result in preferential decomposition of silane on the particle surfaces. The objectives of this phase of the investigation were to:

- Demonstrate that a significant silicon bed/reactor wall temperature difference can be established.

- Demonstrate that silicon sintering can be minimized by proper operating conditions.

- Demonstrate stable electrical performance.

- Identify electrical properties useful for scale-up.

This study was carried out using a 2-inch diameter column shown schematically in Figure 6.38. Nitrogen, preheated to 200°C, was used to fluidize the bed which was about 6 inches deep for more tests. Power was applied to the bed by means of a 0.5 inch diameter center electrode. A majority of the tests were conducted to determine the effect of operating conditions on bed sintering. Tests were performed with metallurgical-grade and semiconductor-grade silicon beds of various mean particle size. Both plain and coated electrodes were evaluated.

CENTER
ELECTRODE

MATCHING
TRANSFORMERS

GUARD HEATERS

DISTRIBUTOR

POWER SUPPLY
1600W AT 50Ω
8K - 120K HZ

GAS PREHEATER

ROTOMETER

N_2

● — THERMOCOUPLE

REACTOR: 2.068 ID
2.375 OD
27 LG.(FLG.TO FLG)

7/8" INSULATION BETWEEN GUARD
HEATERS AND REACTOR WALL
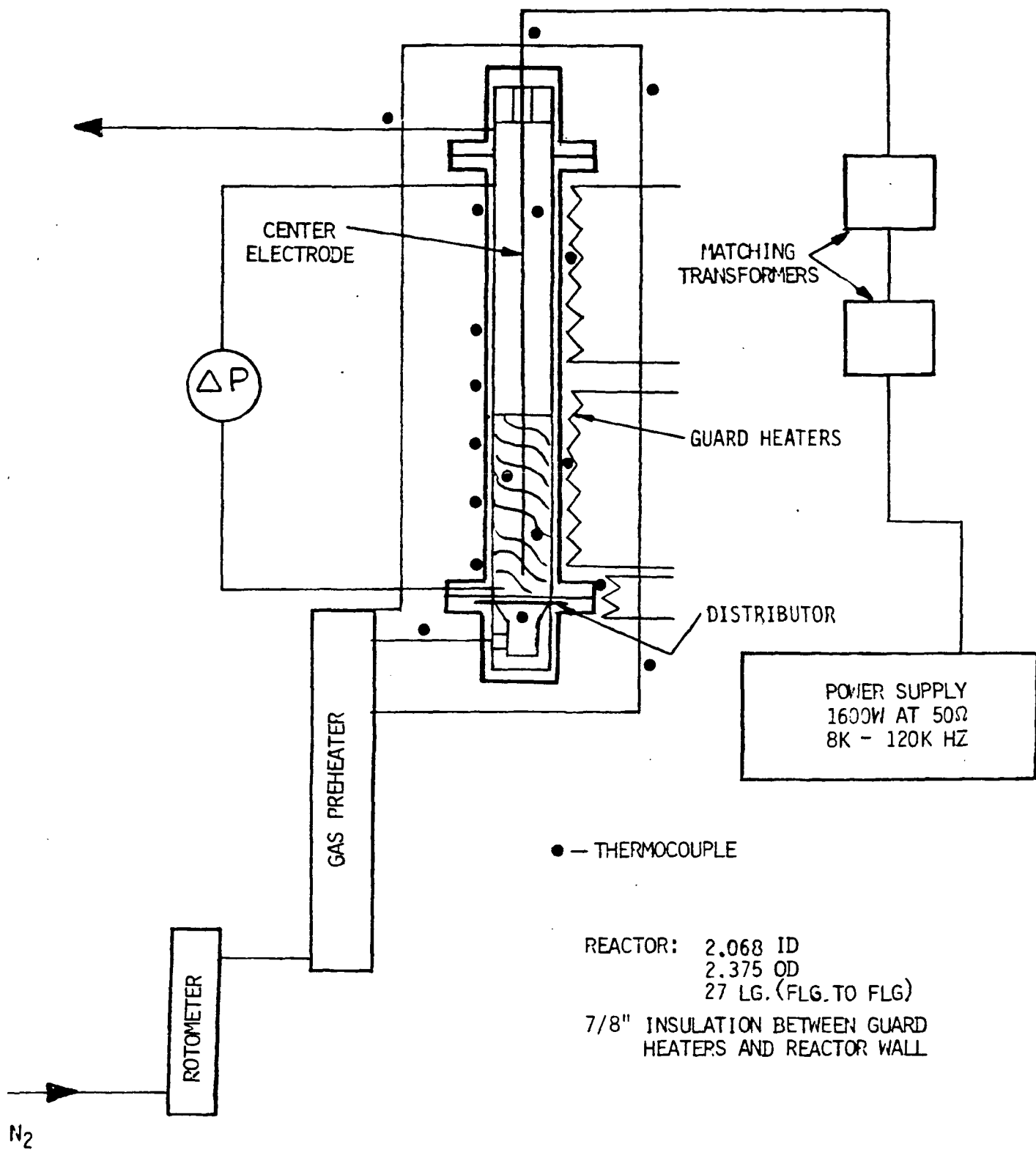
6.38 Capacitive Heating Reactor

Summary

The establishment of a favorable bed/wall temperature difference was demonstrated by capacitively heating a fluidized silicon bed. Bed/wall temperature differences of 26°C, 13°C, and 15°C were measured at heights of 2 inches, 4 inches, and 6 inches in a 2-inch diameter, 8-inch deep bed maintained at 750°C average temperature. This operating condition was achieved at an input power of 200 watts and a low sintering rate of 1% per day.

Particle sintering is a function of the field current density. To reduce current density for a given input power, an electrode geometry is required which will provide a large field area. An optimum operating condition can be achieved by adjusting the spacing between electrodes. While the field area cannot be readily determined, it is inversely proportional to the load impedance and this property may be used to gage the field area. For the 2-inch diameter experimental reactor, a load impedance of less than 8 ohms was necessary to achieve a sintering rate less than 5% per day.

Particle sintering is also a function of the particle size. This was shown in the tests conducted with a metallurgical-grade silicon bed and also with a semiconductor-grade silicon bed of wide particle size distribution. The particle sintering temperature is related to the particle size because the smaller particles are heated more rapidly by the electrical discharge between particles while in the electric field.

The fluidized bed electrical characteristics are mainly determined by the fluidizing media electrical properties. This is because the operating mode for heating is by gaseous discharge from particle to particle in the electric field. While the experimental work was performed with nitrogen, similar performance characteristics are expected for hydrogen.

In the experiments, the capacitively-heated fluidized silicon bed was at least a 90% resistive load as indicated by an average phase angle of 24°. This was supported by oscilloscope observations and by the frequency independence of the load.

There was an indication that a higher bed temperatures and input power, a higher U/U_{mf} is required to achieve stable electrical performance and to control particle sintering in the electric field. The U/U_{mf} increased from 3 to 4 at temperatures up to $500^{O}C$ to 7.5 at $750^{O}C$ in laboratory tests.

Experiments.

a. Bed Heating Tests

The reactor was fabricated from 2-inch, schedule 40, stainless steel pipe and was 27 inches long from flange to flange, as shown schematically in Figure 6.38. The guard heaters were spaced about 7/8 inch from the reactor wall. The space between the wall and guard heaters was filled with ceramic fiber insulation. This arrangement favored the establishment of an optimum temperature gradient from bed to wall to guard. Electrodes were made from 0.5-inch O.D. x 1.065-inch wall stainless steel tubing with a welded cap at the bottom end. Some electrodes were also flame-sprayed with an alumina or silicon coating.

Tests were conducted by establishing an electric field between the centrally located electrode and the reactor wall or between the electrode tip and a porous stainless steel distributor. Tests were also conducted with pyrex and mullite wall liners which terminated 1.5 inches above the distributor plate. A porous quartz distributor was used in some of the tests. These various modifications were used to control the field geometry.

Particle growth due to sintering was measured after each test, except No. 25, by screening the bed through the largest screen size in the original bed. This procedure measured only the particles larger than the largest particles in the original bed. Sintering of small particles to sizes less than the largest original particle size was measured on test No. 25. For this test, which had a sintering rate of 6.2% per day, the size

distribution was measured before and after the test. The smallest particle fraction decreased about twice as much as the rest of the bed. The results were:

Mesh Size	Decrease Per Day	Original Bed Composition
-35/+60	6.8%	47.4%
-60/+80	6.1%	27.6%
-80/+120	11.3%	25.0%

A summary of the capacitive heating tests is presented in Table XII. The results are grouped by silicon grade, metallurgical or semiconductor, and by type of electrode. Within each group tests are arranged in order of increasing sintering.

Bed temperature and electrical data are mean values calculated for the entire test period. For most tests operating conditions varied over a wide range of temperature and power. U/U_{mf}'s are average values for the test period.

In the beginning of the program all tests were performed with metallurgical-grade silicon. The results indicated that the larger the particle size the lower the sintering rate, and a low load impedance resulted in less sintering. Starting with Test No. 12, all tests were performed with semiconductor-grade silicon. Tests No. 12 through 15 were set up to determine the electrical characteristics and sintering rate as a function of a field geometry. To accomplish this, a pyrex wall liner was installed which terminated 1.5 inches above the quartz distributor plate. The bed thermocouple sheathes were coated with a ceramic adhesive for electrical isolation. In this way an electric field could be established between the electrode tip and the exposed reactor wall section. However, the thermocouple coating had a tendency to flake with the result that alternate electrical paths were present in some tests and the effect of field length was not determined. A relationship between load impedance and sintering was determined; this relationship, shown in Figure 6.39, does not apply for the other tests in this group with the exception of Test No. 25 which agrees.

TABLE XII

SUMMARY OF CAPACITIVE HEATING TESTS CONDUCTED IN 2-INCH REACTOR

TYPE OF SILICON	ELECTRODE	TEST NO.	$\overline{dp}$ μ	U/Umf	BED TEMP °C	CAPACITIVE HEATING						SINTERING % PER DAY	ELECTRODE TIP POSITION (Inches)	REMARKS
						TIME HRS.	POWER WATTS	Z Ω	θ DEGREES	E_EFF VOLTS	I_EFF AMPS			
METALLURGICAL GRADE	PLAIN	8	550	3	356	23	110	6.3	39	30	5	2	5/1	114K Hz
		1	375	6	380	81	88	9.9/1.9-/21		28/14	3/7	2	0.6	S.S Distrib., Pyrex Wall Liner
		10	375	6	460	8	105	145	37	138	1	11	5.5	
		7	187	8	408	7	225	15.2	44	69	5	91	5.5	
		2	375	4	454	21	223	0.9	29	15	17	--	0.8	Special disc type electrode
	ALUMINA COATED	9	550	3	365	24	120	4.6	54	31	7	3	2	Filled pores, sanded to reduce thk., 115K Hz
		4	375	6	635	6	90	3.6	66	28	8	51	0.8	as sprayed, 110K Hz filled pores 115K Hz
		6	187	6	400	11	139	32	33	73	2	63	5.3	
	SI COATED	5	375	6	390	8	138	8.5	29	37	4	70	0.8	60K Hz
HIGH PURITY SEMI CONDUCTOR GRADE	PLAIN	13	187	6	420	20	146	3.9	3	24	6	0.2	2.5	Pyrex Wall Liner
		12	375	6	460	23	133	5.7	25	29	5	0.5	1.5	Pyrex Wall Liner, 2.7"bed
		20	223	8	736	45	191	3.7	0	24	7	1	7	8"bed, S.S. Distrib.
		19	221	6	802	21	191	1.6	21	18	11	4	1/4.5	S.S Distrib
		25	222	9	675	47	184	8.2	25	41	5	6	7.3	8.5"bed new silicon,dyna-pore distrib
		15	187	6	450	22	133	8.3	11	36	4	7	5	Pyrex Wall Liner
		14	187	6	450	18	144	8.5	38	40	5	12	3.5	Pyrex Wall Liner
		24	223	18	751	26	450	47	0	123	3	17	6.6	7.6"bed hole in SS Distrib
		18	229	4	837	16	262	3.4	30	32	9	20	2.5	Mullite wall liner, temp pulse >1000°C
	SI COATED	17	187	6	500	24	130	3.1	19	21	7	2	0.5	Wall liner extends to dist no TC's in bed SS dist 110K

NOTES:
1. Bed Temp. and electrical data are mean values for test period
2. 15K Hz frequency
3. Porous quartz distributor plate
4. 6" bed height
5. For test No. 10, 12-15, &18 wall liner stops 1.5" above distributor
6. 0.5" dia S.S. Electrode

The chart shows axes:
- Y-axis: SINTERED PARTICLES, PERCENT/DAY (logarithmic scale from 0.1 to 100.0)
- X-axis: LOAD IMPEDANCE, OHMS (0 to 10)

Text within chart:
125 – 150 WATTS
400 – 500°C TEMP
PLAIN S.S. ELECTRODE
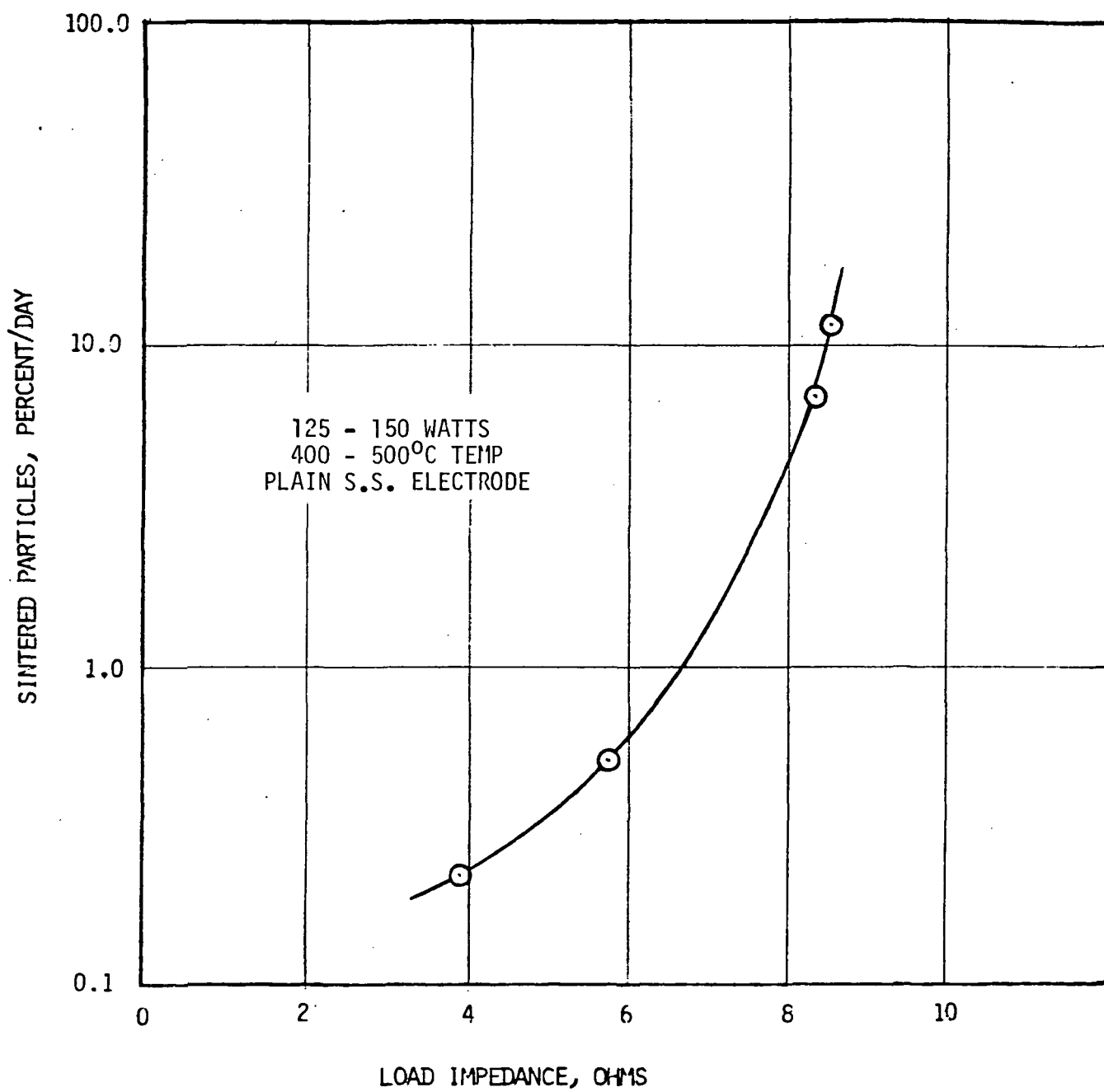
6.39   Capacitive Heating Tests No. 12 – 15 —
       Sintering vs. Load Impedance

To eliminate problems with the thermocouples in the electric field, most of the remaining tests were conducted with deeper beds of about 8 inches. The electrode was positioned near the top of the bed. Tests were conducted at higher temperatures and low load impedance. The higher sintering rate for Test No. 18 was the result of a hot spot in the bed caused by the field between the electrode and a thermocouple sheath. For Test No. 24 the high sintering rate was mainly caused by the abnormally high U/U_{mf} which was required to maintain a fluidized bed with a defective distributor plate.

Matching the load to the power generator was not dependent on the frequency in most cases, an indication that circuit reactance was not large. Tests with plain stainless steel electrodes performed equally well at any frequency from 8K to 115 K hz. Marginal improvement was obtained with the coated electrode tests when operated at high frequency. This would be expected since coating the electrode builds reactance into the circuit.

Based on the results of these tests, it is concluded that the fluidized silicon bed was at least a 90% resistive load as indicated by the average phase angle of 24°.

A U/U_{mf} of 3 to 4 was satisfactory for tests conducted at temperatures up to $500^{\circ}C$. At higher temperatures there was some inconsistency. Test No. 18, with a temperature of $837^{\circ}C$, seemed to operate electrically stable at a $U/U_{mf} = 4$, but a hot spot was present in the field. Test No. 19 ($800^{\circ}C$) was conducted at a $U/U_{mf} = 6$ with considerable difficulty caused by erratic electrical performance. For Test No. 20 the bed height was increased to 8 inches so that the electric field could be established in a region without thermocouples. At the inital U/U_{mf} of 6, erratic electrical operation was experienced. The velocity was increased in increments until stable performance was obtained at U/U_{mf} of 7.5. The test then ran very smoothly until it was discontinued after 45 hours. Test No. 25 was conducted at an average of U/U_{mf} of 9, but there is evidence that the necessity for the higher velocity was the result of leakage around the distributor plate which caused poor fluidization at the center of the bed. The argument for a higher U/U_{mf} at higher temperature is supported by Test No. 20.

A comparison of tests of known field length shows that a relationship exists between the field potential gradient and the bed velocity ratio in the region of the electric field. This is shown in Figure 6.40 based on the average test values. The test numbers are given in parenthesis. The beds were all about the same mean particle size. It may be possible to develop a more accurate relationship using actual test data points. The field potential gradient may also be a function of particle size but there was not enough data available to establish this.

b.   Bed/Wall Temperature Difference

One of the objectives of this program was to demonstrate that the silicon bed could be operated at a temperature higher than the reactor wall. To establish a favorable temperature gradient, the guard heaters were located on a shield spaced about 7/8 inch from the reactor wall. The space was filled with a ceramic fiber insulation. In operation, the shield temperature was adjusted to be approximately $50^{\circ}C$ less than the reactor wall temperature. Capacitive heating was used to increase the bed temperature above that of the reactor wall.

The steady-state temperatures recorded for Test No. 20 are presented in Figure 6.41 which is representative of tests conducted with the electrode at the top of the bed and an input power of about 200 watts. While this geometry was used to avoid the presence of thermocouples in the electric field, it is also recommended as the preferred arrangement for the fluid-bed pyrolysis reactor.

c.   Electrical Characteristics

Startup of the electric field requires going through a transition from high voltage and low current to low voltage and high current to achieve the required power input to the fluid bed. This may be described as a transition from a high impedance to a low impedance load and it increases the requirements of the matching transformer network. As an aid in making this transition, the electrode assembly should include a method of adjusting
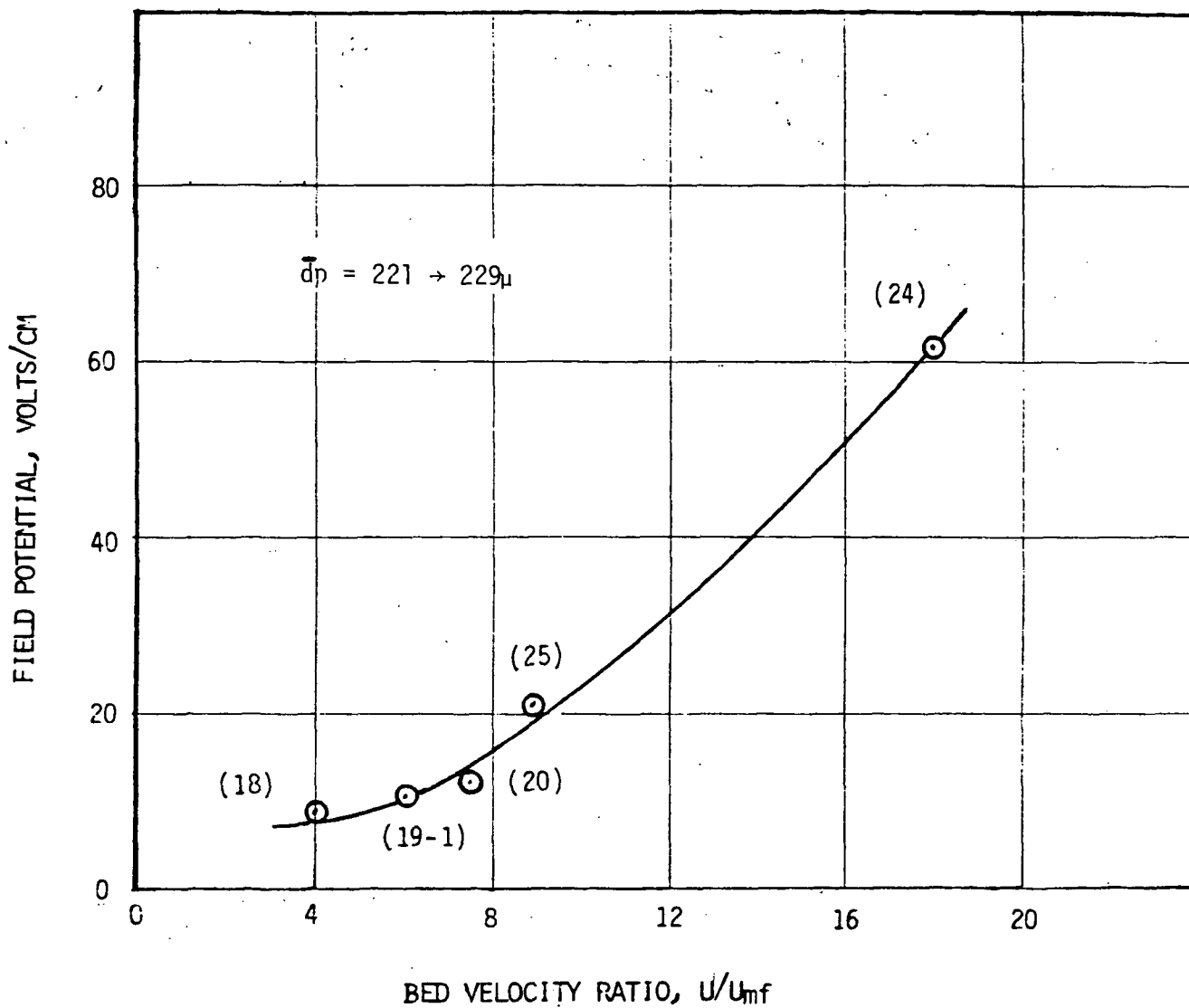
FIGURE 6.40   Field Potential Vs. Bed Velocity Ratio During Capacitive Heating
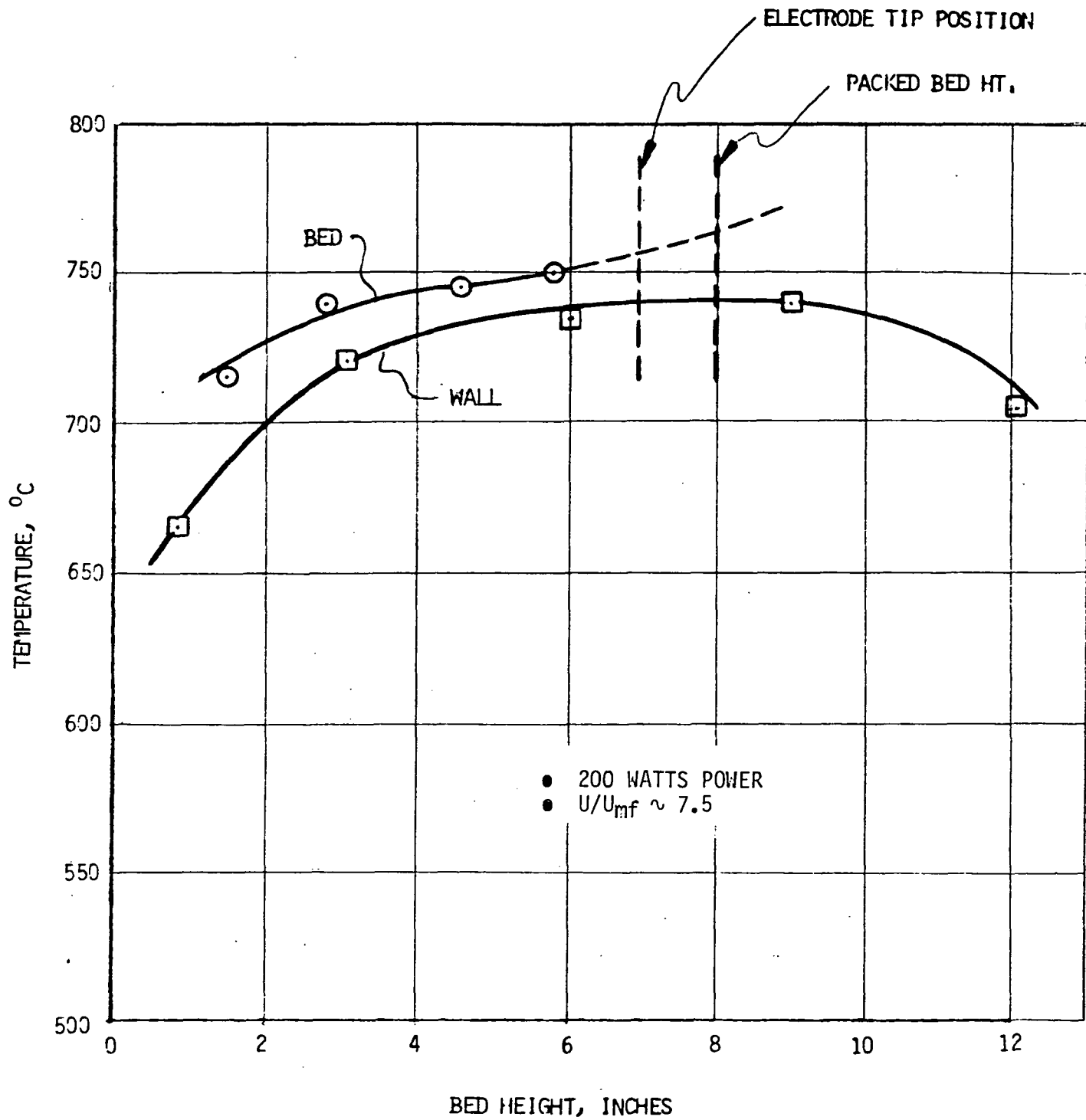
FIGURE 6.41   Wall and Bed Temperatures for Capacitive Heating Test No. 20

the electrode gap so that the field length can be very short at startup. After operating power has been established, the electrode gap can be increased and the matching transformers adjusted to obtain the condition necessary for a low sintering rate.

Test No. 16 was devised to determine the behavior of an electric field in a fluidized silicon bed. The bed thermocouples were removed so that a pyrex wall liner could extend down to a porous stainless steel distributor plate which also served as a ground electrode. The 0.5-inch diameter power electrode was positioned at various heights above the distributor. The test was conducted at two temperatures with the results shown in Figure 6.42. A constant input power of 100 watts was used and the fluidizing media was nitrogen. Each $400^{o}C$ point represents an average of several test measurements at each electrode spacing. For the $20^{o}C$ curve, each point represents a single measurement. The data shows that the field area decreases as the electrode spacing is increased and that there is a great deal of variation in field area during operation. The difference between the two curves is related to the properties of the fluidizing media.

Electrical measurements were also made with a pair of 1-inch diameter stainless steel electrodes in a 6-inch diameter column at room temperature. In this case data were collected as a function of electrode spacing using two different fluidizing media, nitrogen and helium. The gas pressure and velocity were also varied.

The results are shown in Figure 6.43. Data were also collected for U/U_{mf} up to 4, bed penetration up to 24 cm, and power up to 250 watts. The load impedance is very dependent on the fluidizing gas. These results are for the operating condition existing after the transition to low voltage has been attained. As would be expected, the impedance increased with electrode spacing for both gases. For helium, the impedance increased linearly at an average rate of 650 ohm/cm and there was an extreme variation in the field. Nitrogen results indicate a non-linear relationship with a rapid decrease in field area for the first 2 cm of electrode spacing. Also, the field in nitrogen shows less variation at all spacings.

Graph labels:
- Y-axis: LOAD IMPEDANCE, OHMS (values 1, 10, 100)
- X-axis: ELECTRODE SPACING, INCHES (values 0, .5, 1, 1.5, 2, 2.5)
- Curve labels: 20°C, 400°C
- 100 WATTS POWER NITROGEN
- 0.5" DIA SS ELECTRODE
- PYREX LINER
- POROUS SS GROUND ELECTRODE
- 15000 HZ
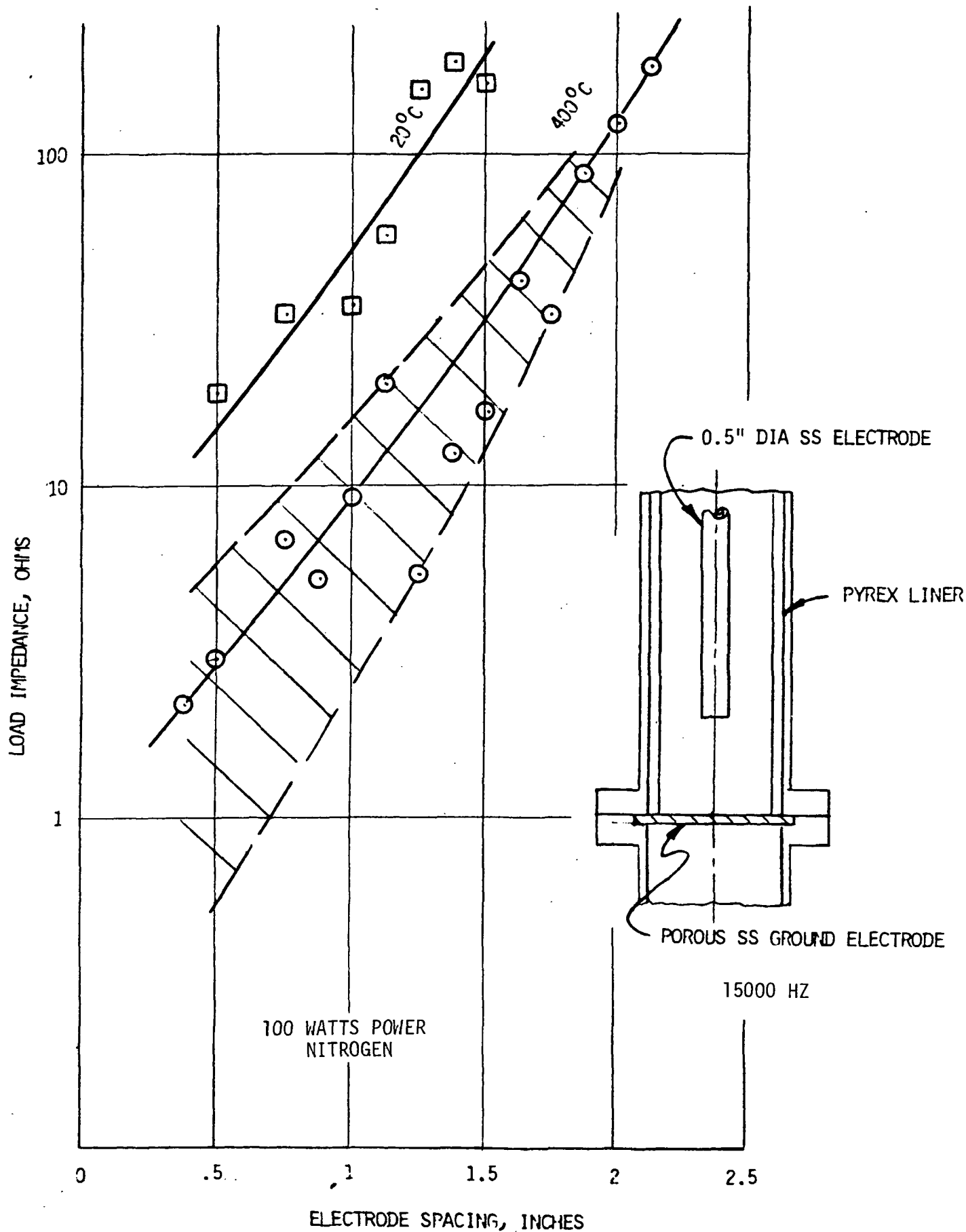
FIGURE 6.42   Test No. 16 -- Electrical Properties Load Impedance
Vs. Electrode Spacing

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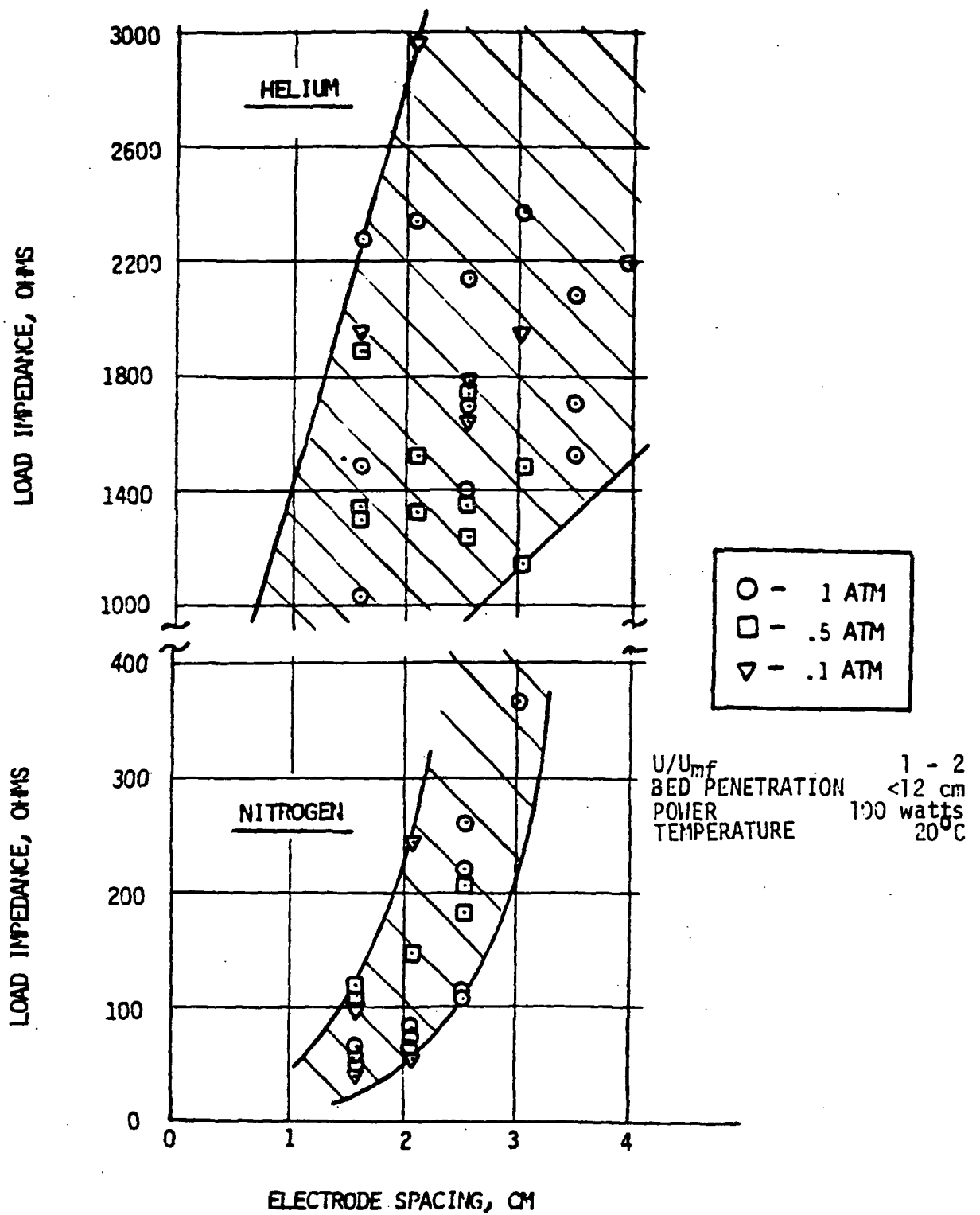
FIGURE 6.43 One-Inch Diameter Electrode Assembly-Load Impedance vs. Electrode Spacing

Tests showed that for a given electrode spacing, increasing the input power decreased the load impedance indicating that the field area may have increased. The change could also be related to an increasing fluidizing gas temperature in the electric field at the higher input power.

With helium it was also possible, under certain conditions, to establish a gaseous discharge between the electrodes above the bed by increasing the power. This could then be extinguished by increasing the electrode penetration into the bed.

For a static bed, once the transition phase to low voltage has been exceeded, a permanent shorted path of sintered silicon particles is formed. Subsequent application of power results in current flow as soon as the voltage is applied.

A pulsating voltage condition is typical when operating with nitrogen. A steady voltage is typical for helium. For both gases, the startup voltage was 4 to 5 times more than the operating voltage. The oscilloscope showed the load amps and voltage to be in phase after the breakdown voltage was exceeded. Before breakdown occurred, a large phase angle was observed.

Recommendations

Two electrode pairs with electrode diameters of 1 inch should be used in the 6-inch diameter fluid-bed pyrolysis reactor. The ground electrode should be designed so that it is offset from its rotational axis. Then by positioning it within a 90° arc, the electrode spacing (field length) may be adjusted with respect to the power electrode. This arrangement plus a matching transformer network with variable taps should permit tuning the load to match the power generator while at the same time optimizing the load impedance to minimize sintering.

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Each electrode assembly should have its own power generator and matching transformer network for independent operation. A series inductor may also be required when using coated electrodes which add circuit capacitive reactance.

It may be possible to satisfactorily heat the fluid bed with one electrode pair. However it's not known if the sintering rate would be acceptable. By having a second electrode circuit available, the input power can be divided and the current density reduced to achieve a lower sintering rate.

1.6.3.2.2   Particle Separation Tests

The goal of the test program was to design a boot located below a fluid bed which will selectively segregate large particles for removal as product. This program was successful and an intra-company Engineering Memorandum was prepared to document the results. A detailed summary of the test program is presented in the paragraphs that follow.

Introduction
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The specific objective of these tests was to develop a technique for preferential withdrawal of coarse particles from a fluid bed of wide particle size distribution. In the current work, the pyrolysis reaction conditions of hydrogen at one atmosphere pressure and 680°C were simulated in a cold model using helium gas at 1/6 atmosphere. The separation of large particles having a mean diameter ratio 2 to 3 times greater than the bed mean size was demonstrated using a pipe of reduced diameters (boot) at the bottom of the 6-inch diameter column. An orifice-type separator was also evaluated and was found to be unsuitable.

Tests were performed with binary particle systems, consisting of 20% to 33% larger particles in a bed of fines. This was followed by tests with beds of wide particle size distribution. Twelve of the thirteen tests

were performed with fluid beds which were reduced in both volume and mean particle size as silicon was extracted from the boot. For the final tests, the bed was maintained at a near constant size by periodically adding silicon, previously removed from the boot, to the column. In the final test, the preferred coarse particle size was 51% to 61% of the bed.

Conclusions

Separation of coarse silicon in binary particle systems is possible for coarse/bed particle diameter ratios of 2.55 and greater. Separation was not possible for a diameter ratio of 2.3 or less. Continuous separation of coarse silicon from a bed of wide particle size distribution was demonstrated using a 1.5-inch diameter boot separator (Figure 6.44).

The velocity ratios were identified for a separator with a 1.5-inch diameter; these ratios covered a column U/U_{mf} range from 1.1 to 2.75 to simulate hydrogen at one atmosphere at $680^{o}C$ (Figure 6.45) and also a column U/U_{mf} for 1.1 to 2.25 to simulate hydrogen at 1.44 atmosphere at $20^{o}C$.

Tests conducted with the terminal velocity orifice separator were not successful and the method has been rejected for this application.

Discussion

A schematic of the particle separation column is shown in Figure 6.46. With this arrangement, fluidizing gas may be introduced at a transition cone through jets, and at the bottom of the boot section through a porous metal distributor; this permits velocity adjustments over a wide operating range. The column was designed for vacuum operation with the pressure controlled by means of a manual throttle valve and vacuum pump. Boot silicon samples, ranging from 300 to 500 grams, could be withdrawn through a tube at the bottom without interrupting the fluidization of the column. The samples were graded using ASTM screens and a RO-Tap shaker. The vacuum hopper shown was added

- Helium at 0.17 ATM
- 50 Lb. Silicon Bed, Wide Particle Size Distribution
- Constant Bed Volume Maintained
- 1.5" Diameter Boot Separator

Lost Separation Temporarily

U/Umf = 1.6-2.2

U_{Boot} = 0.59 Ft/Sec

$\overline{dp}$ = 210 – 178 μ

3.14 Lb/Hr

U/Umf = 2.2-2.3

U_{Boot} = 0.81 Ft/Sec

AVG $\overline{dp}$ = 178 μ

2.79 Lb/Hr

U/Umf = 2.7

U_{Boot} = 0.82 Ft/Sec

AVG $\overline{dp}$ = 176 μ

2.53 Lb/Hr

Boot/Bed Particle Diameter Ratio

Fluidization Time, Hours
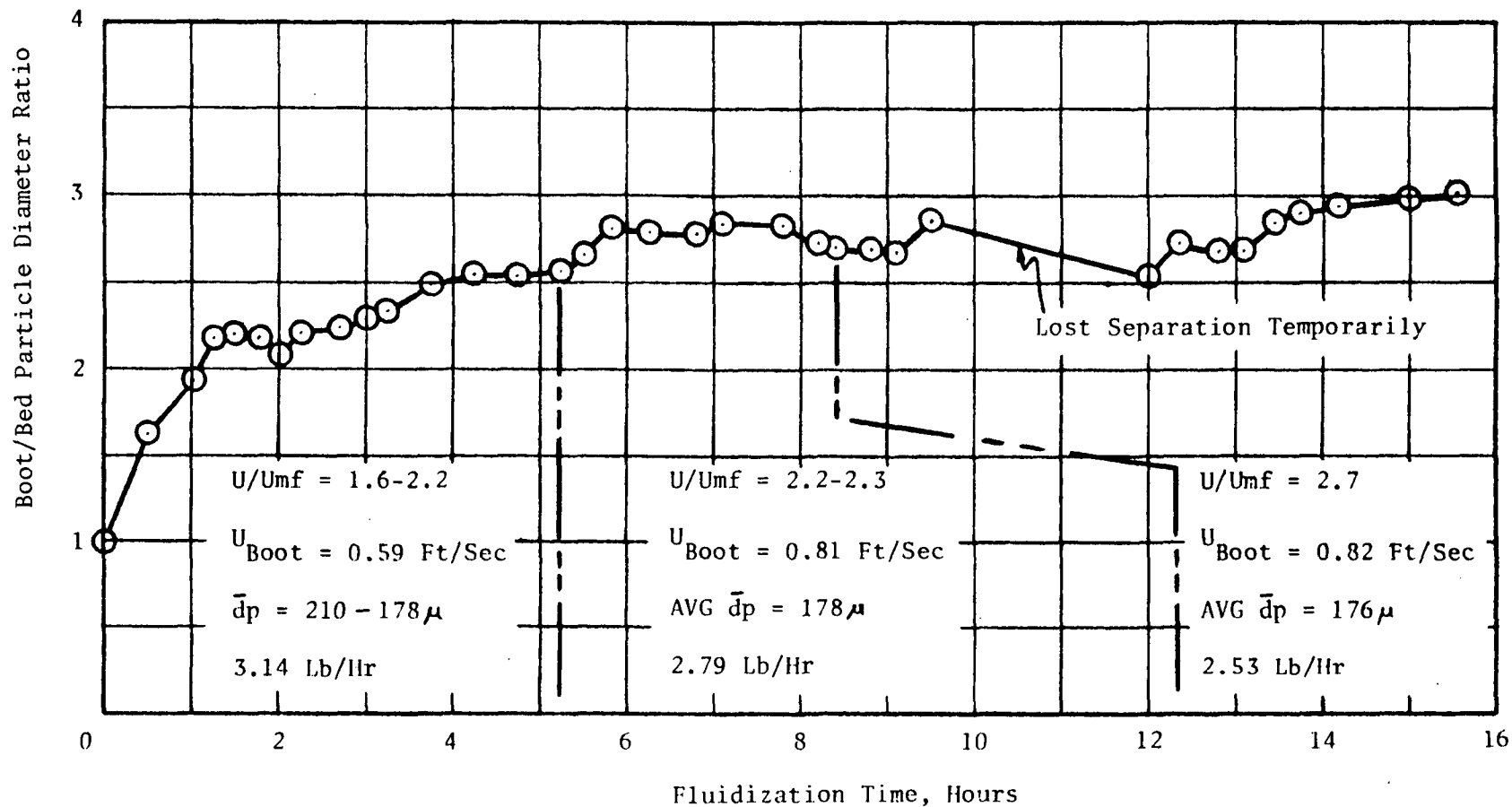
FIGURE 6.44   Test No. 13 - Separation Diameter Ration Vs. Fluidization Time 1.5" Diameter Boot

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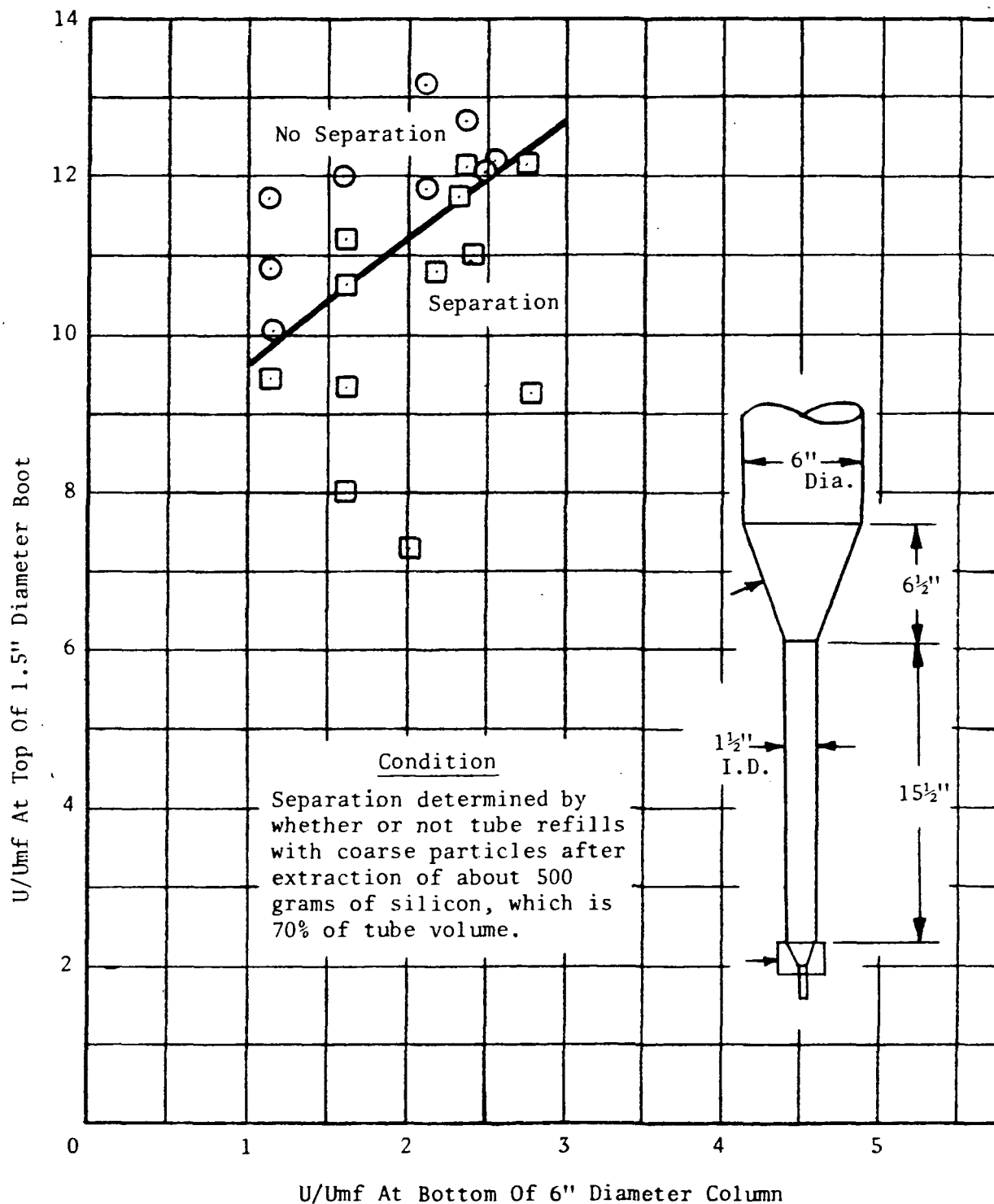
FIGURE 6.45   Velocity Ratios for Particle Separation with 1.5" Diameter Boot
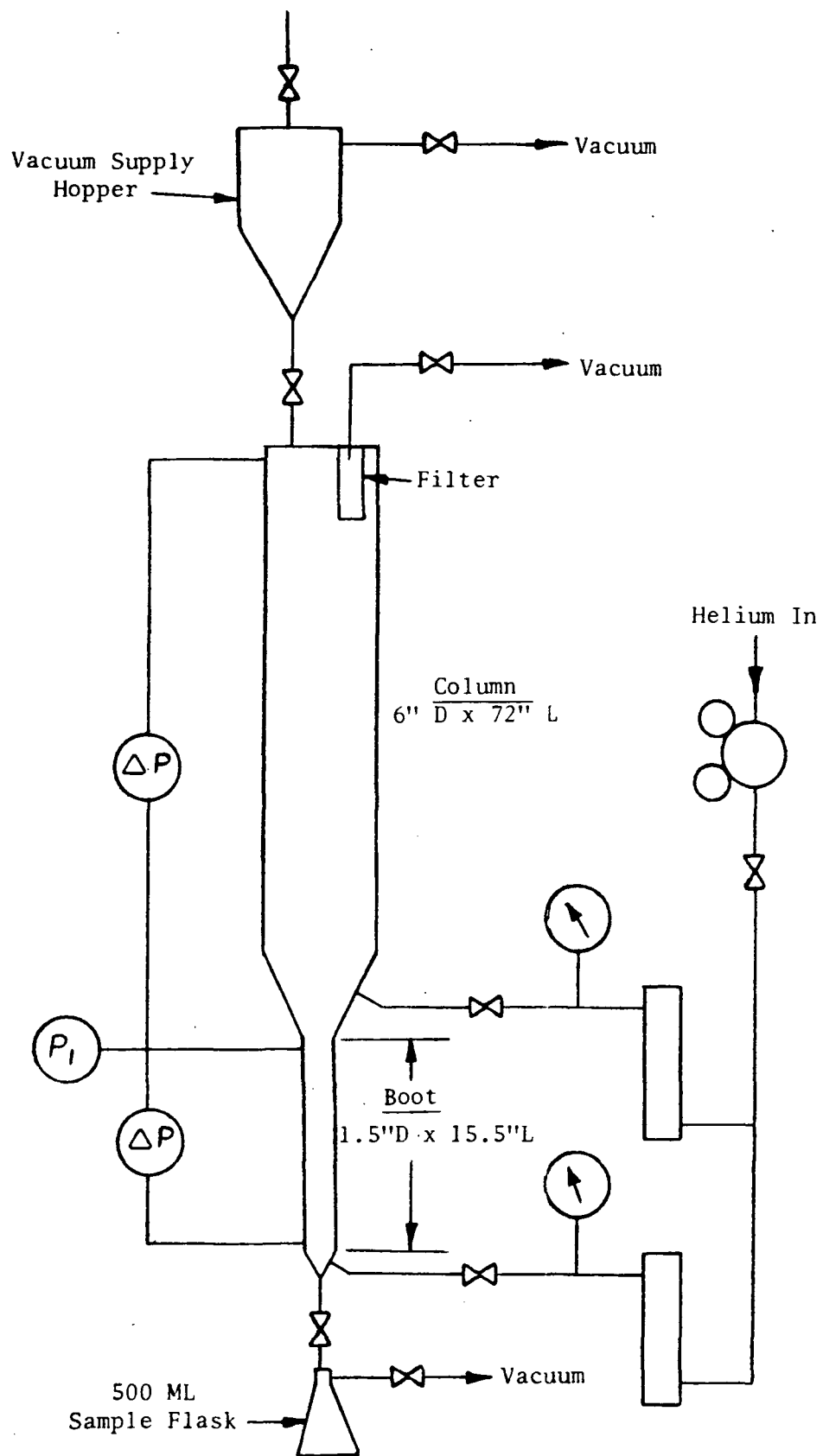
FIGURE 6.46   Particle Separation Test Column Schematic Diagram

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for test No. 13 so that the bed could be maintained at constant volume by periodically replacing silicon without interrupting column fluidization.

The various boot geometries studied are illustrated in Figure 6.47. Initial tests with binary particle systems were performed with a 3-inch diameter boot. This was followed by tests with beds of wide particle size distribution using the 2-inch diameter boot separator and the orifice separator. Two orifice diameters, 7/16-inch and 1/4-inch were evaluated. Only marginal separation was achieved with the orifice separator. While the average orifice velocity may be high, the instantaneous velocity varies over a wide range allowing perculating of bed silicon through the orifice. Tests with the 2-inch diameter boot separator were encouraging, and seemed to indicate that additional improvement may be achieved by further reduction in the boot diameter.

An operational disadvantage of these tests was the reduction in bed volume, and in mean particle size over the test period. This made the demonstration of a continuous separation process difficult. Therefore, before continuing the tests with a 1.5-inch diameter boot separator, a vacuum hopper was installed at the top of the column so that silicon could be added periodically without interrupting the column fluidization. The procedure used was to add 1000 grams to the column after the removal of two 500-gram samples from the boot. Initially, the first 10 pounds of silicon added to the bed had the same composition as the bed at the beginning of the test. After this quantity, samples removed from the boot were cycled back to the bed. The average bed composition was known at all times during the test period with the bed weight maintained between 48 and 50 pounds.

Another serious limitation in the tests was that the fluid bed conditions in the boot could not be observed. To correct this problem, the 1.5-inch diameter boot separator was fabricated from clear polycarbonate tubing with the transition cone being adapted from a 1000 ML transluscent plastic vacuum flask. The separator dimensions are shown in Figure 6.47.

The results of the separation tests using the 1.5-inch diameter boot, presented in Figure 6.44, show that continuous separation was demonstrated

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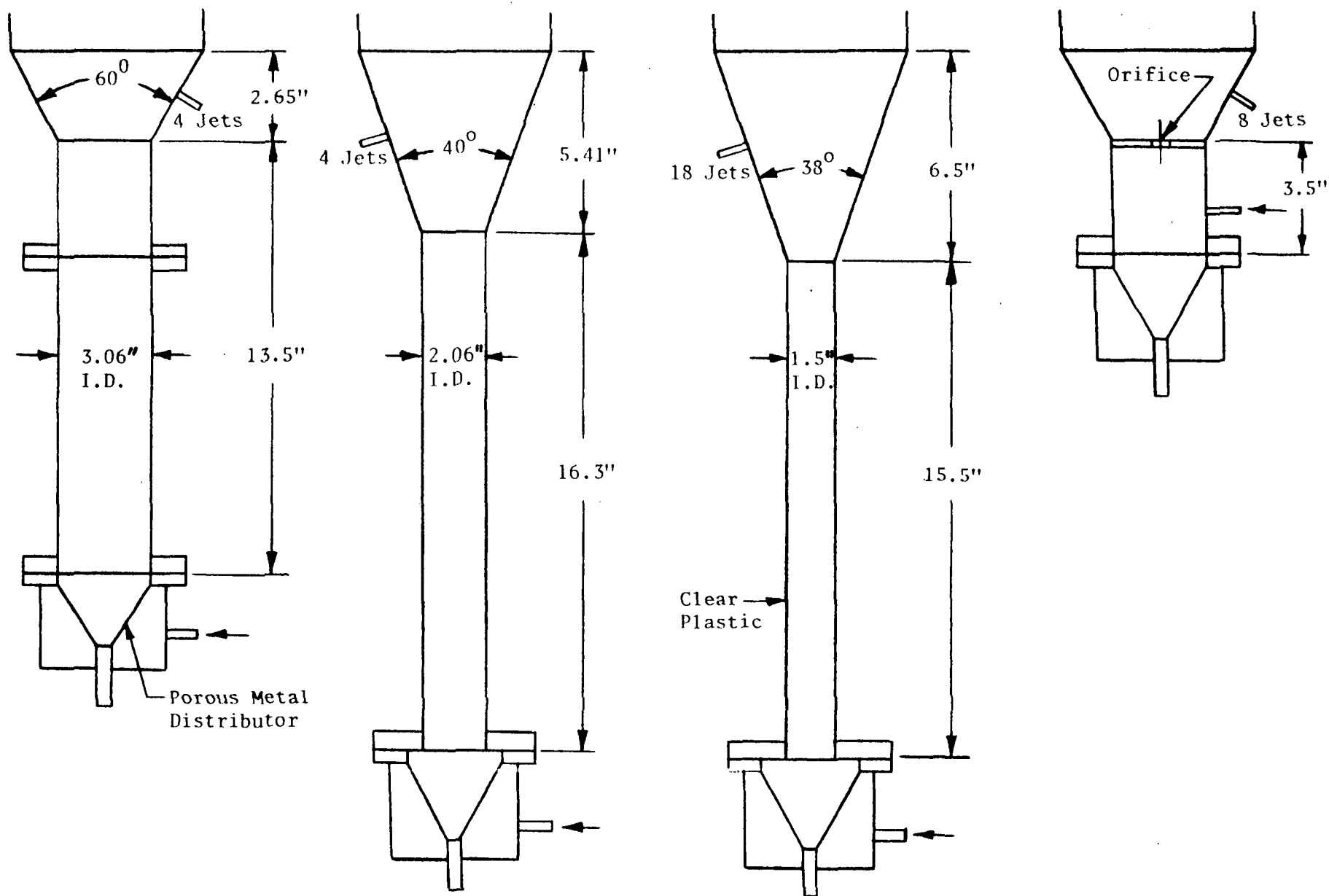
FIGURE 6.47   Boot Particle Separator Configuration

over a 15.5-hour test period with only one loss of separation occurring as indicated on the curve. This came about because the test was stopped immediately after emptying the boot of coarse particles. Restarting at the previous velocity resulted in a mixing type of slugging flow in the boot involving the entire boot length. To re-establish separation it was necessary to reduce the velocity at the top of the boot to a U/U_{mf} = 2.5 for the bed mean particle size. Then, as the boot refilled with coarse silicon, the velocity was gradually increased to its operational value. Over two hours were required in this instance to recharge the boot with coarse silicon. For later tests, in which operating conditions were mapped, about one-half hour was the typical time required.

Interrupting the test immediately after removal of coarse silicon changes the flow condition occurring in the boot. Normally a slugging flow is created at the top of the boot when smaller size silicon fills in to replace material extracted. The quantity of coarse silicon removed determines the distance of the slugging flow in the tube. This slugging is a plug type with sections up to 2 inches long that do not include the coarse silicon at the bottom of the tube. This type of flow results in the separation of fines, and a distinct coarse/mixed particle interface which progresses up the tube as coarse silicon is separated. Withdrawing more than 70% of the coarse silicon in the tube can also result in a mixing type of slugging flow involving the entire tube length with no separation occurring.

Test No. 13 can be divided into three sections representing different operating conditions. In the first section, the bed mean particle size decreased because the bed was replaced with new silicon of the original composition. After this material was used, samples removed at the beginning of the test were cycled back into the bed, and the bed particle size stabilized. During this period, the U/U_{mf} increased from 1.6 to 2.2. For the second section, the same column U/U_{mf} was used, but the boot velocity was increased. At the top of the boot the U/U_{mf} = 11.5 for the bed mean particle size. In the third section, the boot velocity remained the same, but the column U/U_{mf} was increased to 2.7. Particle separation was achieved for all three conditions. The increasing velocity resulted in some increase in the boot mean particle size. However, the time required for the boot to refill with coarse

silicon was much greater at the higher velocity as shown by the decreasing separation rates noted in lb/hr for each section. The third section excludes 2 hours to compensate for the period when separation was lost. Typical boot refilling times were respectively, six, eight, and fourteen minutes. Since the boot velocities were the same in sections two and three, the almost doubling of the refilling time appears to be caused by higher U/U_{mf} at the bottom of the column. An average sample mass of 500 grams was obtained from this test.

Tests were also conducted to determine the operating conditions which will result in loss of separation after withdrawing 500 grams of coarse silicon from the boot. These tests were conducted with helium and nitrogen, and are equivalent to fluidization with hydrogen at 1 atm and 680°C, and hydrogen at 1.44 atm and 20°C. Differences both in gas density and viscosity are included in calculating the equivalent performance. The results presented in Figure 6.45 for the helium case are applicable for the stated condition only. It is believed that this condition represents the maximum quantity that should be withdrawn for this boot geometry. Reducing the quantity extracted to 50% or less of the tube volume should shift these demarcation curves upward because the tendency towards a mixing type of slugging is reduced by the increased amount of coarse silicon remaining in the boot tube permitting an increase in velocity. The nitrogen tests indicate that the boot velocity must be reduced when increasing the U/U_{mf} in the column, while the helium tests indicate this is not necessary. An explanation for the difference in performance cannot be given at this time.

As a final test, the bed was depleted over the test period, using the 1.5-inch diameter boot separator. The objective was to demonstrate continuous separation for this operating condition. Over a period of 17.5 hours the bed volume was reduced from 47.5 lb with a $\bar{d}p = 185\mu$ to 32.1 lb and a $\bar{d}p = 128\mu$. The column U/U_{mf} was maintained at 2 by periodically adjusting the flow to compensate for the decreasing bed particle size. As the bed decreased in particle size, it was necessary to decrease the boot velocity from an initial 0.75 ft/sec (9.4 U/U_{mf}) to 0.30 ft/sec (6.0 U/U_{mf}) at the end of the test in order to prevent a mixing type of slugging when a sample was withdrawn. Samples averaged 400 grams, and were 95% or more

coarse fraction particles. The average separation rate was 0.9 lb/hr whereas for test No. 13, the rate was 2.5 lb/hr.

Visual observation of the flow conditions in the boot was necessary for the success of this test.

Recommendations

On the basis of these tests, a 1.5-inch diameter boot, as shown in Figure 6.48 is recommended for the 6-inch diameter fluid-bed pyrolysis reactor. This is similar to the plastic boot tested, except the tube length has been increased to 20 inches to compensate for the reduced volume of the porous cone distributor. Five hundred grams of silicon, $\bar{d}p = 500\mu$, is equal to 65% of the tube volume, and is the largest quantity that should be withdrawn at one time.

Three pressure taps are indicated. By measuring the pressure difference across two half lengths, the fluid bed operating conditions can be more accurately ascertained. When coarse silicon is extracted from the bottom, the U/U_{mf} at the top of the boot increases as the silicon particle size in that region decreases. This results in a fluctuation Δp measurement across the upper tube increasing in amplitude. With the start of Δp fluctuations in the lower tube, the extraction should be terminated. As the tube refills with coarse silicon, and the flow changes from one of a slugging bed to minimum fluidization, the upper tube Δp measurement becomes stable indicating that another quantity of silicon may be withdrawn.

To avoid initiating a mixing type of slugging flow in the entire boot, the boot gas flow should not be interrupted immediately following extraction of coarse silicon. Restarting at the operational velocity may result in a mixing type of flow. If mixing is occurring in the boot tube, indicated by similar fluctuating Δp measurements in lower and upper halves, the velocity should be reduced to a U/U_{mf} of about 1.5 at the boot bottom. As coarse silicon starts collecting, the velocity can be slowly increased. In laboratory tests about one-half hour was required to attain operational velocity. A mixing type of flow, once started in the boot, did not show any tendency to self damp in laboratory tests.

6" PS Flange
ASA 150 Lb
Welding Neck

6-3/4" Dia. Holes,
60° Apart

1" Dia. Disc of
Dynapore Fluid-
izing Media
Welded Over Each
Hole

1.5"

4.5"

6.5"

3-3/4" Dia. Holes,
120° Apart

1"

Pressure Tap, 3

9"

20"

1½ I.D. Tubing

18"

60° Cone
Dynapore Fluidizing
Media

1/2" I.D. Tubing
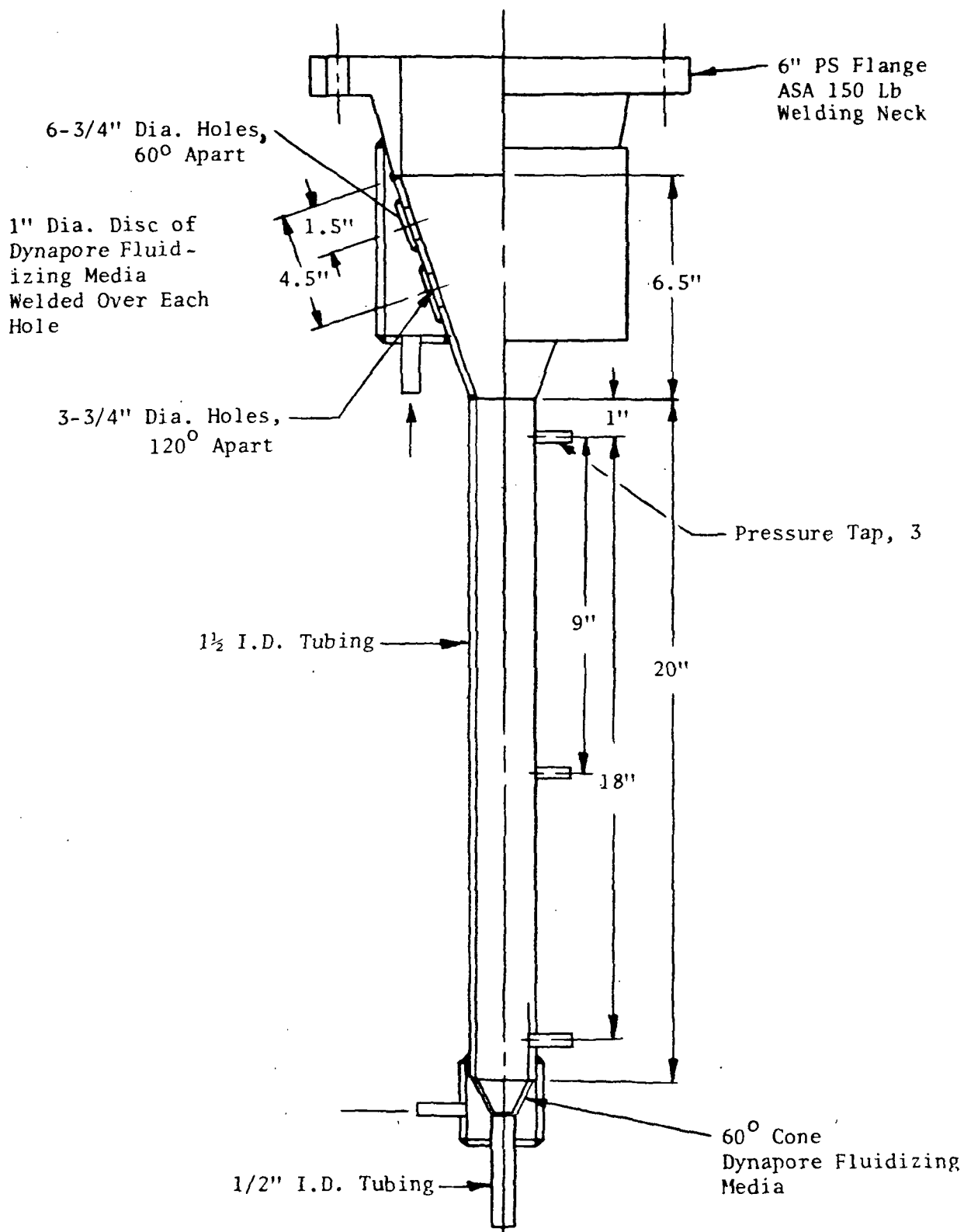
FIGURE 6.48   Boot Particle Separator Recommended Design for 6" Reactor

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1.6.3.3   Evaluation · Fixed Bed/Fluidization

This effort consists of the analysis of data from the fixed-bed deposition (1.6.3.1) and inert gas fluidization (1.6.3.2) experiments for applicability for designing the fluid bed pyrolysis PDU. The data analysis resulted in the establishment of the base case conditions for the design of the 6-inch diameter fluid bed PDU.

Silane feed concentration	10%
Bed temperature	$650^{\circ}C$
Mean particle diameter	250
U/U_{mf}	4
Boot diameter	1.5 inch
Starter bed	Acid washed & etched silicon

It is expected that under these conditions, decomposition of silane in the fluid bed will be predominantly heterogeneous and decomposition morphology will be dense and coherent.

1.6.3.4   Fluid Bed Pyrolysis (PDU)

This work item consists of all efforts associated with the design, fabrication, and testing of an experimental fluid-bed to establish design data for an EPSDU-scale system.

The fluid bed silane pyrolysis reactor is an attractive alternative to the free-space reactor for decomposing silane. A silane-hydrogen gas mixture is flowed through a heated fluidized bed of silicon particles. Heterogeneous decomposition occurs on the silicon particle surfaces and the particles grow up to about 1/3 of a millimeter in size. To allow for a continuous process, small seed silicon particles are periodically added to the fluid bed while the fully-grown particles are withdrawn through a particle separation boot. The advantages of the fluid bed are:

- Silane pyrolysis/consolidation is performed in one step

- The product is free flowing and easily handled

- Power consumption is small and the process is economically feasible if over 10% silane is fed at 630°C.

The results and conclusions from the early work (fixed-bed work and inert gas fluidized bed testing) indicated that pyrolysis of silane in a fluid bed should be successful. A fluid bed design and fabrication was completed in 1980. By January 1981, installation was completed and after checkout, the unit was ready for operation in April 1981.

This report describes the results of seven runs performed in April and May during which arc heating was attempted, silane added to the fluid bed and silicon deposited on fluid-bed particles. Due to budgetary constraints, the PDU test program was not completed and the unit was temporarily shut down in May 1981. The program is scheduled to continue in fiscal year 1982 if adequate funding becomes available.

Summary

1. The successful startup of the fluid bed pyrolysis unit was demonstrated for silane feed composition up to 21%, fluid bed temperature of 540 to 685°C and bed velocities from 0.6 to 1.2 ft/sec (U/U_{mf} = 4.5 to 7). No problems were encountered with agglomeration or plugging of the fluid bed particles. Powder formation was minimal and the morphology of the silicon coating on the bed particles was good.

2. The temperature of the porous metal distributor cone is critical to the operation of the fluid bed unit. During silane addition, its temperature rose from 400 to 525°C. At these temperatures, silane pyrolysis is occurring on the distributor and deposited silicon eventually plugged the distributor plate. The distributor plate temperature must be kept well below 400°C for long-term operation (changes will be incorporated during the continuation of the program).

3. Almost complete silane conversion (99.7%) was achieved when the fluid bed was 640 to 685°C and silane feed composition as high as 21% for a fluid bed height of 3 ft.

4. During the two silane runs of about 6 hrs, many process parameters were changed, such as bed temperatures and silane feed composition so that no long-term steady-state runs were made. Also, for longer runs, product withdrawal and seed additions would have been required to sustain continuous operation. Neither of these operations were attempted in the silane runs and continuous operation still remains to be demonstrated.

5. Arc heating was attempted in a silicon bed fluidized with hydrogen. Difficulties were encountered in sustaining operation longer than 2 hours. However, while operational, arc heating did prove to be an effective method of additional bed heating and creating higher bed temperatures than the reactor walls. Arc heating was not attempted with a silane feed. The results of the arc heating tests show that more development work is required before this mode of heating can operate for long lengths of time.

6. Providing heat to the fluid bed through the reactor walls with electrical band heaters did not prove detrimental to fluid bed pyrolysis operation. Reactor walls showed no signs of excessive coating or wear. However, an operating time of 6 hours was too short to reach a conclusion on this type of heating.

PDU Description

A P & I diagram for the fluid bed process development unit (PDU) is shown in Figure 6.49. Figure 6.50 shows the main features of the PDU. These are (1) a particle separation boot of 1½-inch diameter through which the fully-grown particles are removed, (2) a fluid bed reactor of 6-inch diameter where silicon particles are grown and heated, and (3) an expanded head of 18-inch diameter to prevent elutriation of the smaller particles. Before the effluent gases are vented, they pass through a 2-micron filter which traps the powder made by silane decomposition.

Silane and hydrogen are blended in a process stream (Figure 6.49) and then passed through a porous metal cone distributor to provide fluidization. As the silane heats, it decomposes into silicon and hydrogen and the silicon is deposited on the fluid bed particles. The superficial gas velocity in the fluid bed (U) is many times the minimum fluidization velocity (U_{mf}). In the present series of tests, U/U_{mf} varied from 4 to 8. The purpose of the high velocity is to prevent agglomeration of the particles and also to effectively stir the bed material. Particle movement is essential, otherwise particle growth rate will not be uniform and continuous operation of the PDU depends on this parameter.

Hydrogen gas in the boot fluidizes particles at $U/U_{mf} = 8$. This prevents entry of particles smaller than 350 microns in diameter. When the boot fills with these particles, about half of the boot volume is withdrawn and collected in a product hopper. To make the process continuous and maintain constant bed height, small silicon seed particles of about 85 microns are added to the top of the boot and blown into the fluid bed where they will continue to grow.
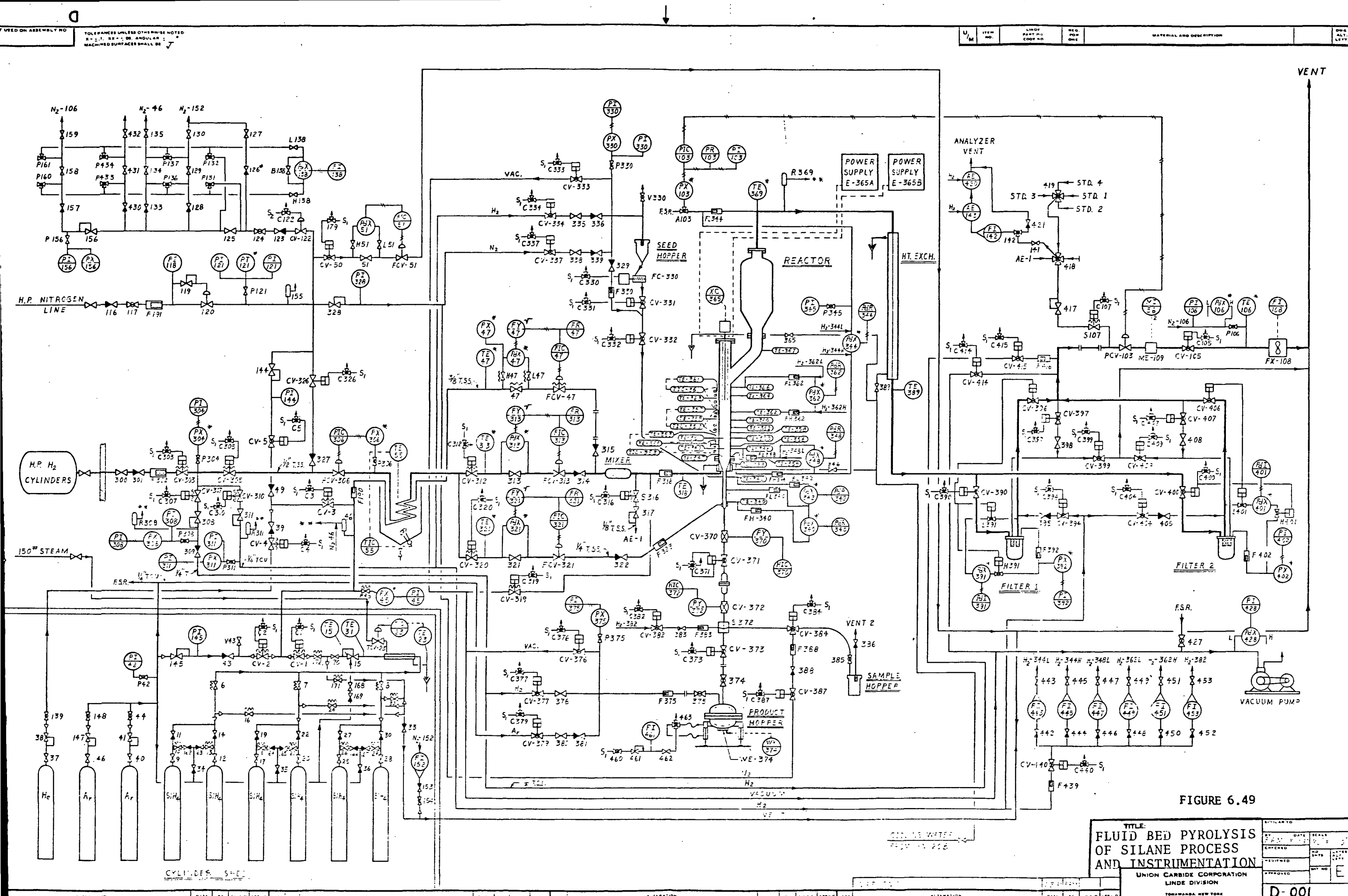
FIGURE 6.49

FLUID BED PYROLYSIS OF SILANE PROCESS AND INSTRUMENTATION

Union Carbide Corporation
Linde Division
Tonawanda, New York

D-001

232/233

FIGURE 6.50 Fluid Bed Reactor Assembly

Discussion

Powder Formation

Some powder or dust was made during silane decomposition Runs 5 and 6. At the end of Run 6, the filter hopper was removed and 53 grams was collected. This amounts to 1.6% of the silicon made by pyrolysis of silane and is, thus a very small fraction. Powder formation was manifested by increasing pressure drop across the porous metal filter. This is shown in Fig. 6.51. During Run 5 when the bed temperature and silane feed composition were low, there was no increase of filter ΔP with time. However, when the feed increased to 10% silane and the fluid bed was hotter, there was a ΔP increase with a slope of 20 inches of water per hour. This indicates the formation of powder. As shown in Figure 6.51, powder was made all during Run 6; the slope was 24 inches of water per hour. It is interesting to compare the results of Runs 5 and 6 with the critical silane concentration curve determined in the fixed bed work. This is given in Figure 6.52. The high fluid bed temperature part of Run 6 lies entirely in the homogeneously decomposition region of Figure 6.52, but powder seems to be made whenever the silane composition exceeded 10% as evidenced by the filter pressure drop curves in Figure 6.51. The amount of powder made is still at an acceptable level.

Silicon Mass Balance

The fluid bed silicon particles were weighed before and after silane addition.

> 42.4 lb before silane addition
> 49.6 lb after silane addition (including wt. of powder
>   collected in filter)
> ─────────────────────────────────
> 7.2 lb silicon net gain

The amount of silane that entered the bed was measured. Since the effluent silane composition was determined by a thermocouple cell, the amount of silane leaving the fluid bed reactor was calculated. The results are shown below:

> Run 5   Silane into fluid bed = 26.68 SCF (70°F, 1 atm)
>   Silane out fluid bed   = 9.05 SCF
>   ────────────────────────────────
>   Silane Reacted   17.62 SCF

235
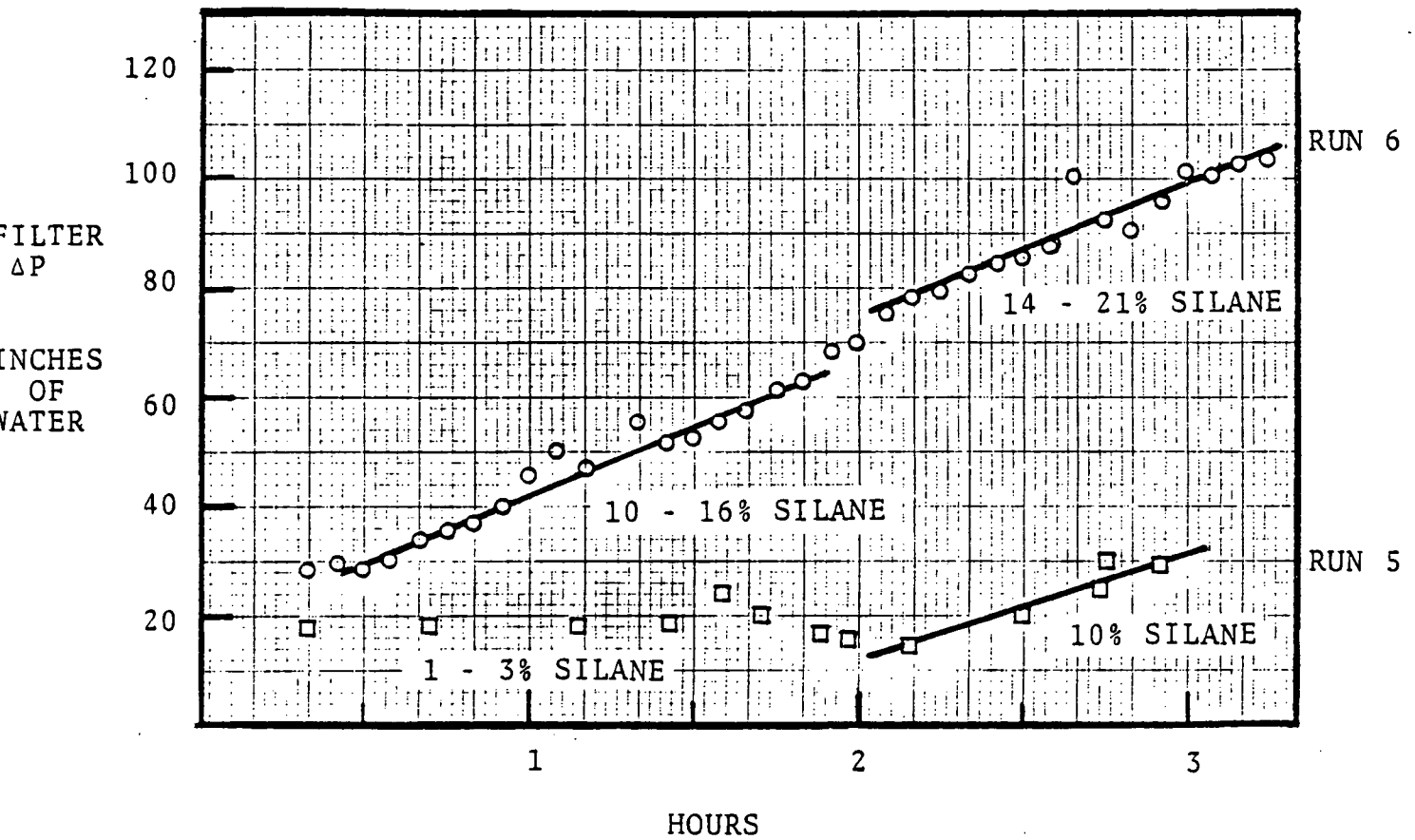
FILTER PRESSURE DROP.   RUNS 5 & 6   SERIES A

Figure 6.51
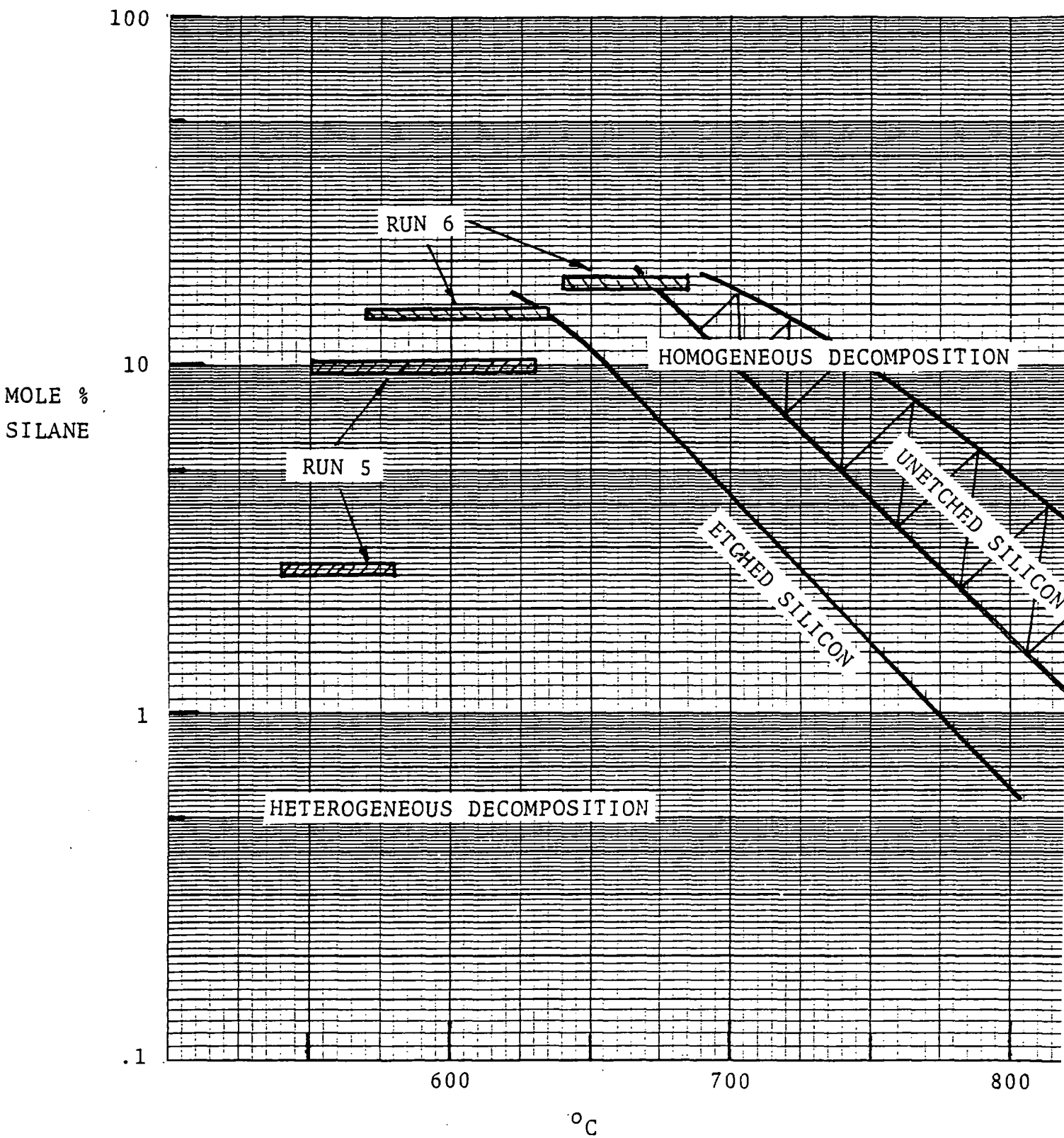
CRITICAL SILANE CONCENTRATION CURVE

Figure 6.52

Run 6   Silane into fluid bed =   86.55 SCF

Silane out fluid bed   =   3.20 SCF

Silane Reacted   =   83.35 SCF

Silicon made   =   6.05 lb

Total silicon made   =   7.33 lb
from Runs 5 & 6

The calculated total of silicon made compares very well with the measured amount of silicon gained in the fluid bed.

Bulk Density and Void Fraction

The silicon particle bed bulk density can be calculated when the bed length and weight are measured. During Runs 5 and 6, the expanded fluid bed height was calculated by means of the fluid bed pressure drop readings. The void fraction, ε, i   ,und by the following relationship.

$$\varepsilon = 1 \qquad \frac{}{}$$

ρ_B = bulk density

ρ_S = silicon particle density, 145.5 lb/ft^3

Run No.	Bed Weight lb.	Bed Height inches	Bulk Density lb/ft^3	ε	
2	44.8	30.0	81.2	.44	settled bed after Run 2
5	42.4	29.5	78.0	.46	poured bed before Run 5
6	49.6	31.8	85.5	.41	settled bed after Run 6
6	43.7	33.6 (calc)	71.6	.51	fluidized bed before silane addition
6	49.6	36.0 (calc)	76.3	.48	fluidized bed at end of silane addition

Silicon Particle Micrographs

Scanning electron microscope (SEM) micrographs were taken for the fluid bed silicon particles before and after silane addition. These are shown in Figures 6.53 and 6.54. A high magnification of the silicon deposit shows a nodular type of growth; some of the smaller nodules approaching 0.2 μm. There appears to be good adhesion of the coating to the silicon particle surface.

A sample of the material trapped in the filter after Run 4 is shown in Figure 6.55. It contains the debris material seen on the particle surface as in Figure 6.53 and possibly dust created by particle attrition. In Figure 6.56 the filter material has an entirely different appearance. This is the result of powder formation during silane addition. Most of the powder is formed in balls from 1 to 15 μm with the smaller particles having sizes around 0.1 μm.

In order to measure the thickness of the silicon coating on the fluid bed particles, a sample of the bed material after Run 6 was mounted, polished and then etched with a HF and nitric acid solution. The results are seen in Figure 6.57. It is encouraging that the silicon coating (from SEM pictures) appears uniform on all the particles, regardless of particle size. This indicates a constant growth rate. Figure 6.58 is a higher magnification of the silicon deposit which is measured to be 5 to 7 μm on the micrograph.

The thickness of the silicon deposit can be estimated with the following relationship:

$$\ell = \frac{W_{Si}}{W_B} \quad \frac{D_p \phi_s}{6} \qquad \text{where } W_{Si} = \text{weight of silicon deposited}$$
$$W_B = \text{weight of bed}$$

$$\ell = 4.9 \text{ μm} \qquad D_p = 273 \text{ μm}$$
$$W_{Si} = 7.2 \text{ lb} \qquad \phi_s = .63^{(2)}$$
$$W_B = 42.4 \text{ lb}$$

This is close to the measured value of 5 to 7 μm.

50X
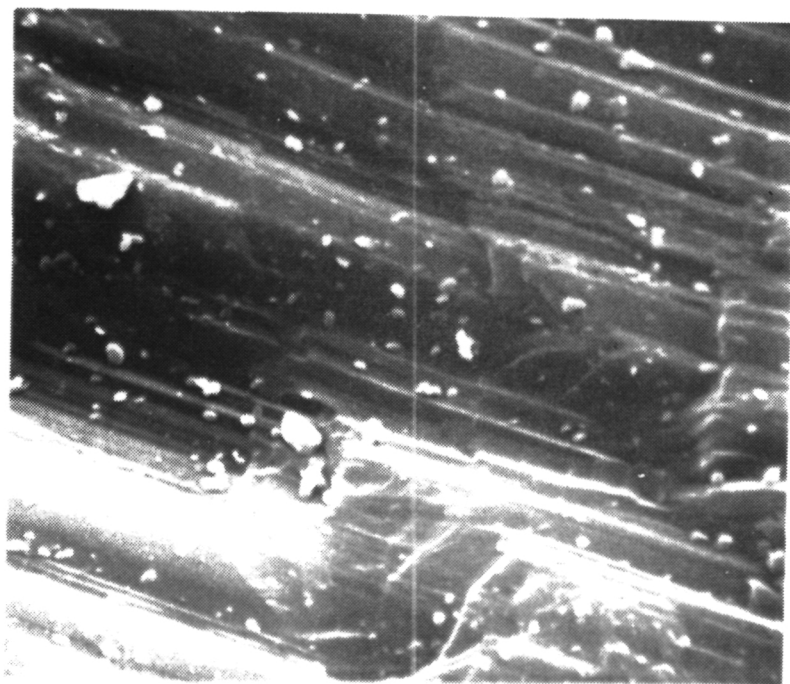
5000X

SEM MICROGRAPH OF SILICON PARTICLES BEFORE

SILANE ADDITION.   RUN 4 SERIES A

Figure 6.53

50X
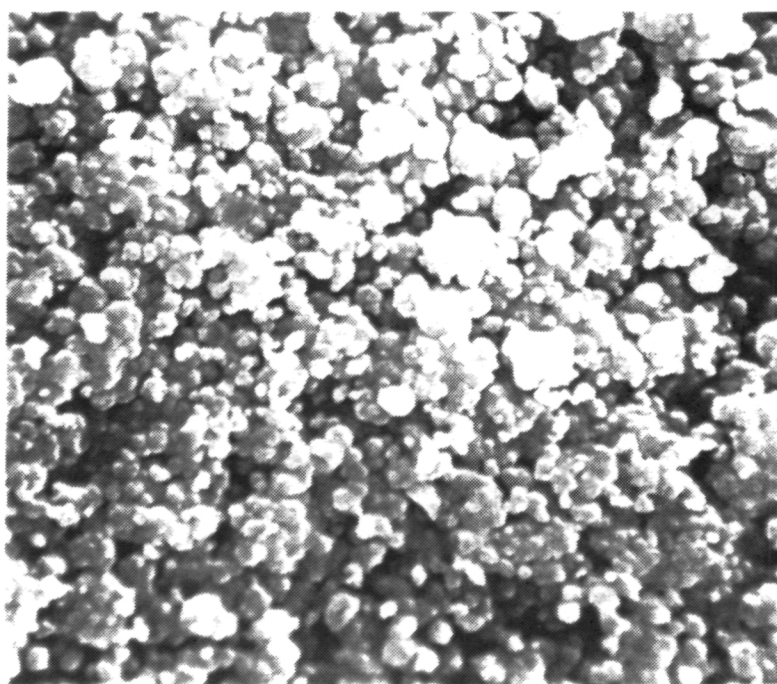
5000X

SEM MICROGRAPH OF SILICON PARTICLES AFTER

SILANE ADDITION.   RUN 6 SERIES A

Figure 6.54
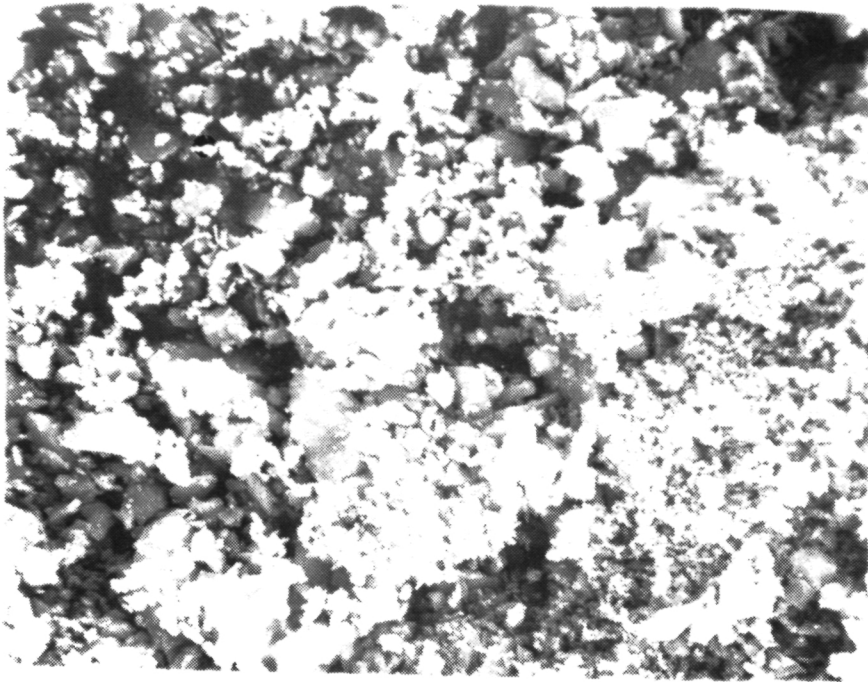
1000X
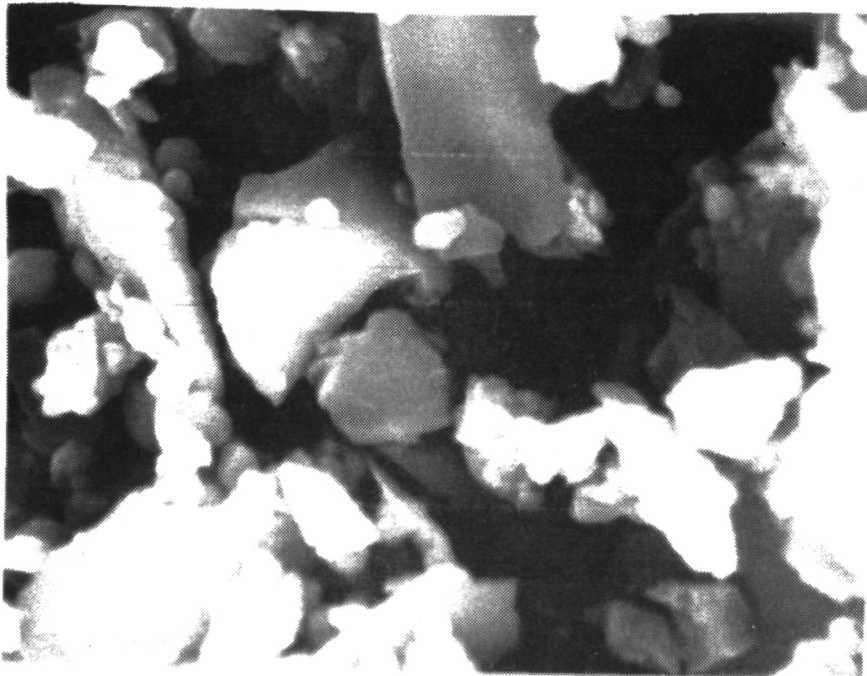
10,000X

SEM OF FILTER MATERIAL AFTER

RUN 4 SERIES A

Figure 6.55
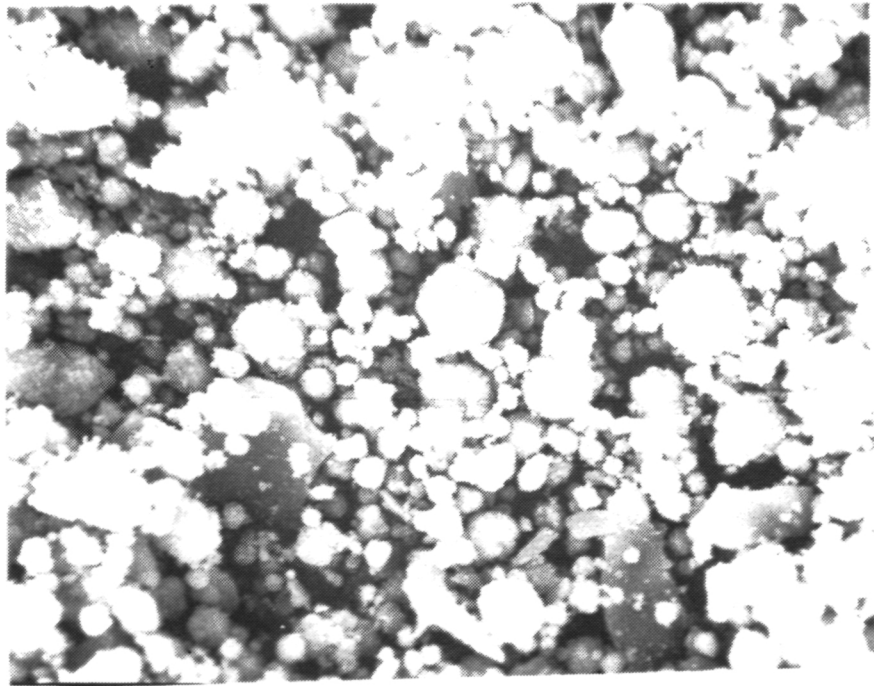
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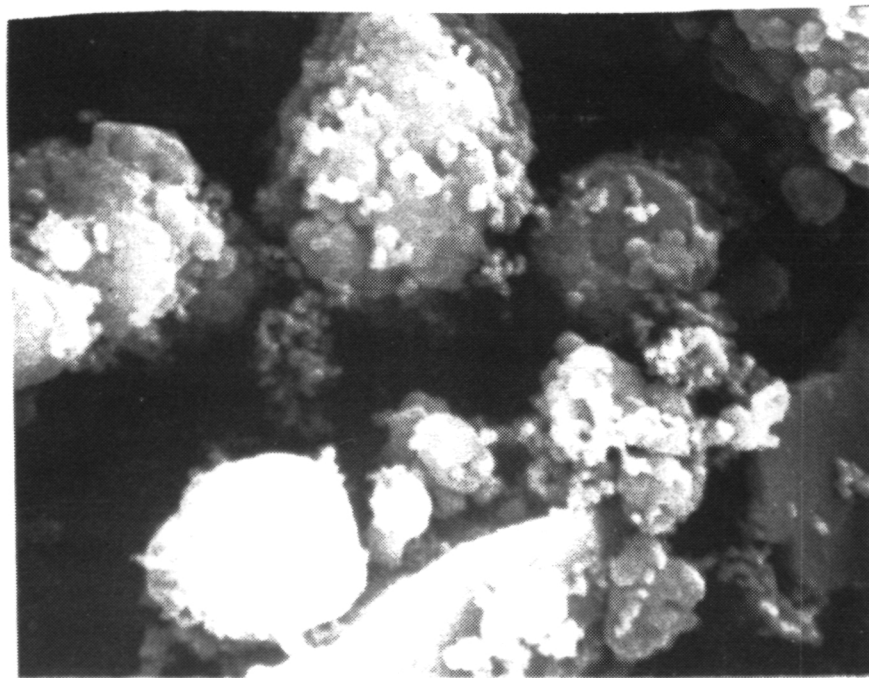
10,000X

SEM OF FILTER MATERIAL AFTER

RUN 6 SERIES A

Figure 6.56

243
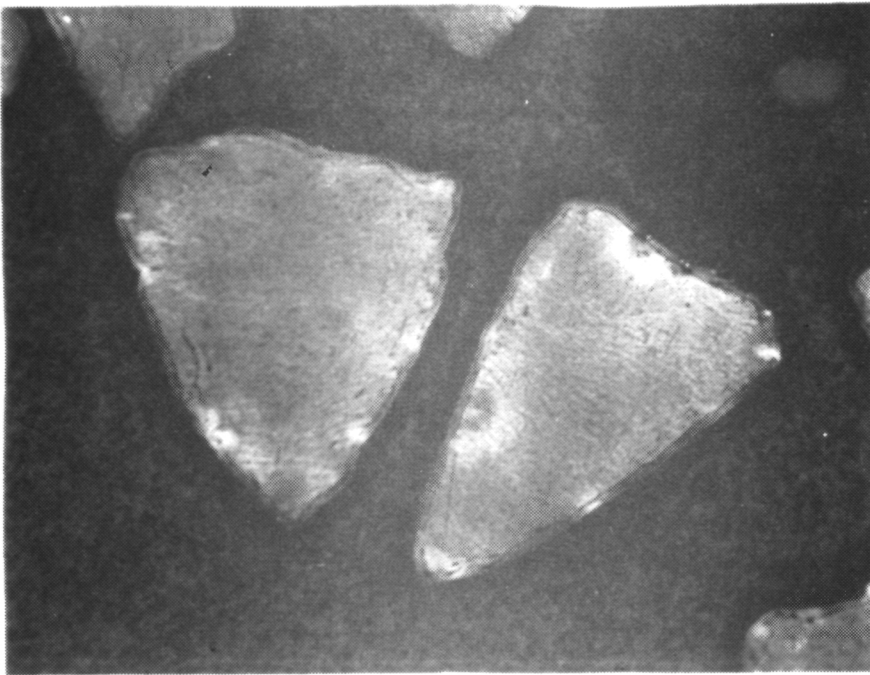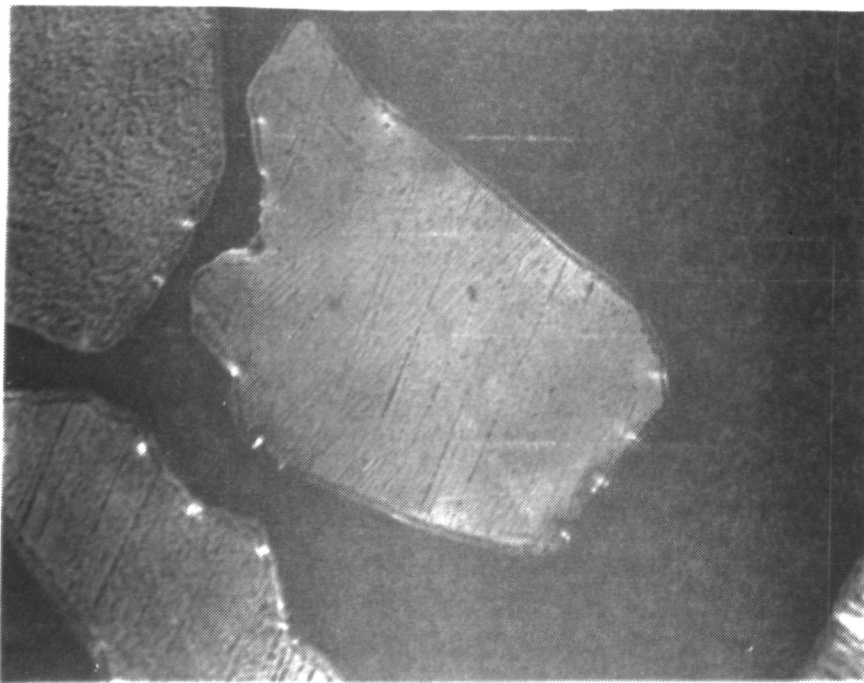
200X

200X

SILICON COATED PARTICLES MOUNTED, POLISHED

AND ETCHED.   RUN 6   SERIES A

Figure 6.57

244

SILICON
LAYER
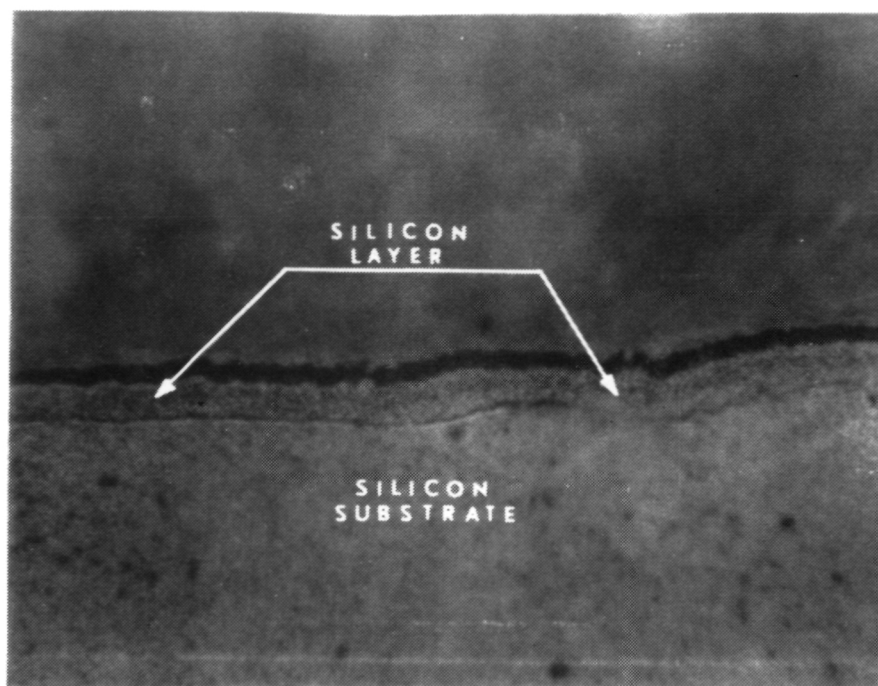
SILICON
SUBSTRATE

ETCHED SILICON PARTICLE SHOWING UNIFORM

SILICON DEPOSITION LAYER

1000X

Figure 6.58

Silicon Particle Growth Rate

The particle growth rate is assumed to be constant and equal to:

$$\frac{\partial r}{\partial t} = \frac{R}{\rho_p A_T}$$

r = particle radius

t = time

R = silicon production rate, lb/hr

ρ_p = silicon density, lb/ft^3

A_T = total surface area of bed, ft^2

It can be shown that:

$$A_T = \frac{6\,W_B}{D_p \phi \rho_p}$$

where W_B = total weight of silicon bed

D_p = diameter of initial silicon particles

ϕ = particle shape factor

The average bed surface area is 3362 ft^2. The average silicon production rate is 1.26 lb/hr. Therefore,

$$\frac{\partial r}{\partial t} = 0.8 \ \mu m/hr$$

The measured silicon coating averages 6 μm. Over a 5.7 hr silane feed time, the actual average growth rate is 1.1 μm/hr. Since the silane addition was not performed at a steady-state condition, the growth rate varied during the runs as the silane concentration and flow were changed. Therefore, the calculated and measured growth rate numbers, shown only for comparison, are reasonably close.

Conclusions

1. Electrical heating bands placed on the outside of a heat shield were found ineffective in heating the fluid bed. The highest temperature attained was only $350^{\circ}C$ and the bed was not uniformly heated.

2. The heat shield was removed and new band heaters were placed on the outside wall of the 6" reactor pipe. This type of heating was successful in reaching a bed temperature of $685^{\circ}C$.

3. Arc heating was tried in two runs. This mode of heating was effective in raising the fluid bed temperature from 50 to $100^{\circ}C$ over its initial bed temperature by the application of 400 to 1600 watts of high frequency power to a pair of electrodes immersed in the fluid bed. Arc heating also created bed temperatures that were 10 to $40^{\circ}C$ warmer than the reactor walls. In both of the above respects, arc heating produced the desired results but it had many deficiencies:

- It was difficult to initiate the arc. It was found that the gas velocity in the fluid bed had to be reduced considerably and the electrodes clean and almost touching.

- After the arc was on, a power inbalance in the load and power supply resulted in unstable operation.

- The stainless steel electrodes were severely pitted after only a few hours of operation.

- A quartz liner, which surrounds the electrodes to provide isolation from ground, was found broken after each test.

- Large sintered particles were found when arc heating was operated at a U/U_{mf} of 2.5, some as large as ½ inch. Less sintering was found when the gas velocity was increased to a U/U_{mf} of 5.

- Arc heating was only tested with a hydrogen environment. It still is not known how silane will affect electrode operation.

4. In two tests silane was added to a fluid bed heated only by external band heaters. Overall, the runs were successful for the conditions used in the tests.

- There was no plugging or agglomeration of the silicon particles in the fluid bed.

- Silane feed was on for a total time of 5-2/3 hours.

- The upper bed temperature ranged from 575 to 685°C.

- The silane feed concentrations ranged from 1 to 21%.

- The superficial gas bed velocity varied from 0.6 to 1.2 ft/sec which corresponds to a U/U_{mf} of 4.5 to 7.0.

- The conversion of silane into silicon was from 57 to 99.7%.

- The silicon production rate was calculated to be from 0.2 to 2.1 lb of silicon per hour.

- SEM micrographs of the coated silicon particles showed good morphology.

- The inner walls of the fluid bed reactor pipe were seen to be clean and free of any deposits.

5. There was a good silicon mass balance. The amount of silane used in the reaction was calculated from silane flow rate data. The quantity of silane in the effluent gas stream was subtracted and the net silane converted amounted to 7.3 lb of silicon. The silicon fluid bed particles were weighed before and after the silane addition and the bed weight gain was 7.2 lb.

6. Some powder was made at the higher bed temperature but still small in quantity. The power collected in the filter hopper amounted to 1.6% of the silicon mode.

7. The temperatures recorded by a thermocouple contacting the distributor cone showed high temperatures. At the start of silane addition, temperatures were around 400°C, eventually reaching 525°C at the end of the run. This caused silane decomposition and a gradual rise in distributor pressure drop was noted during the tests leading to run shutdown.

Recommendations

1. A modified design of the distributor cone is required that can provide adequate cooling to keep its temperature below 400°C. A water-cooling jacket was provided in the present design, but it proved insufficient.

2. A new type of electrical heater is required which is rated for continuous operation at 750°C. Although the band heaters used in the present experiment were rated for 650°C, they were used well above the rating.

3. The temperature control on the reactor wall was divided into three zones; top, middle, and bottom with each zone regulated by a temperature controller. More zones are required, especially at the bottom of the reactor. This may help control lower bed temperatures and in turn, provide lower distributor temperatures.

4. Additional cooling of the 6-inch flanges holding the distributor may be an alternative method of providing lower distributor temperatures.

5. Another method of providing direct heat to the top of the fluid bed may be by the use of an immersion heater. Some types are available which operate at 1300°C.

6. It is possible to add 2 feet to the fluid bed length by installing a flanged spool piece on the bottom of the reactor. This section could be operated much cooler than the upper present section of fluid bed; i.e., 300 - 350°C and help maintain lower distributor temperatures. The disadvantage is that the present product sampling line that exists on the unit must be removed.

7. Some of the pressure taps into the fluid bed plugged after many hours of silane addition. Some bed material was found in these lines. At present, a flow of hydrogen in these lines acts as a purge. Either the purge flows must be increased or some other method must be used to prevent plugging.

1.6.4   QUALITY CONTROL

This task included all activities associated with the development of quality control equipment, techniques, and procedures for use during EPSDU operations.

For control of silane and silicon quality in the EPSDU, analytical tests were developed for detection of trace quantities of harmful contaminants. These tests included methods for detecting boron, phosphorous, and hydrocarbons in silane; improved and consistent rapid electrical tests of silane purity by epitaxy; and bulk deposition of silicon on a seed rod followed by float-zone leveling and resistivity profiling.

A review was conducted of the stream analyses required during EPSDU operation for data collection. These analytical requirements are in addition to the tests to be performed on the silane and silicon products for purposes of quality control. Based on this review, a schedule was developed listing the analyses required, streams to be sampled, and equipment and supplies necessary to perform the required tests. A total of 66 individual sample points were identified. To provide mass balance and reaction kinetic data on the reactors and the distillation columns, six points per unit of equipment were provided with a sample volume buffer so that all six samples may be taken at one time and analyzed sequentially. The system will allow small representative samples to be taken while minimizing material loss and handling difficulties with highly reactive materials.

From the list of required analysis, existing analytical methods were identified which were most appropriate and from this list groupings were made to minimize the number of equipment items required. Finally, a proposed equipment list was developed and a laboratory layout was prepared. Basically, all major components of all streams will be analyzed by a gas chromatograph. Trace levels of gas in silane or other non-chloride containing gases will be analyzed on a second chromatograph. Liquid and solid silicon will be analyzed for low ppm levels of metallic impurities in flameless atomic absorption spectroscopy. Traces of boron and phosphorus in chlorosilanes to sub-ppb levels will be analyzed by UCC's proprietary wet chemical method, using a UV spectrometer. A silicon flake will be analyzed by melting in a high-purity thin-wall quartz crucible and measuring the resistivity of the cast pellet. The slim-rod reactor, epitaxial reactor, and float zone refiner are incorporated into the facility for the determination of the silane and product silicon quality. A core drill has been included in the equipment list so that samples can be taken from the product ingot and zone refined prior to electrical measurements. The preliminary laboratory layout, Figure 6.59, shows an arrangement with a separate center section for the float-zone refiner.

1.6.4.1   Silane Analysis – Slim Rod Deposition

The purpose of the slim-rod reactor is to monitor the purity of silane by pyrolyzing the silane and depositing the silicon onto high-quality silicon rods, 9 mm in diameter x 140 mm long, from a 2 mm square silicon seed rod in less than 8 operating hours. The rod is suitable for float zoning and electrical characterization.

1.6.4.1.1   Design Criteria

The most important criterion in designing the slim rod reactor is its ability to quickly assess silane purity in a routine manner by a moderately skilled technician. The silicon deposition time must be minimized since it can be a very time-consuming operation. This indicates that a small seed rod is preferred over a large one due to essentially a fixed linear growth rate

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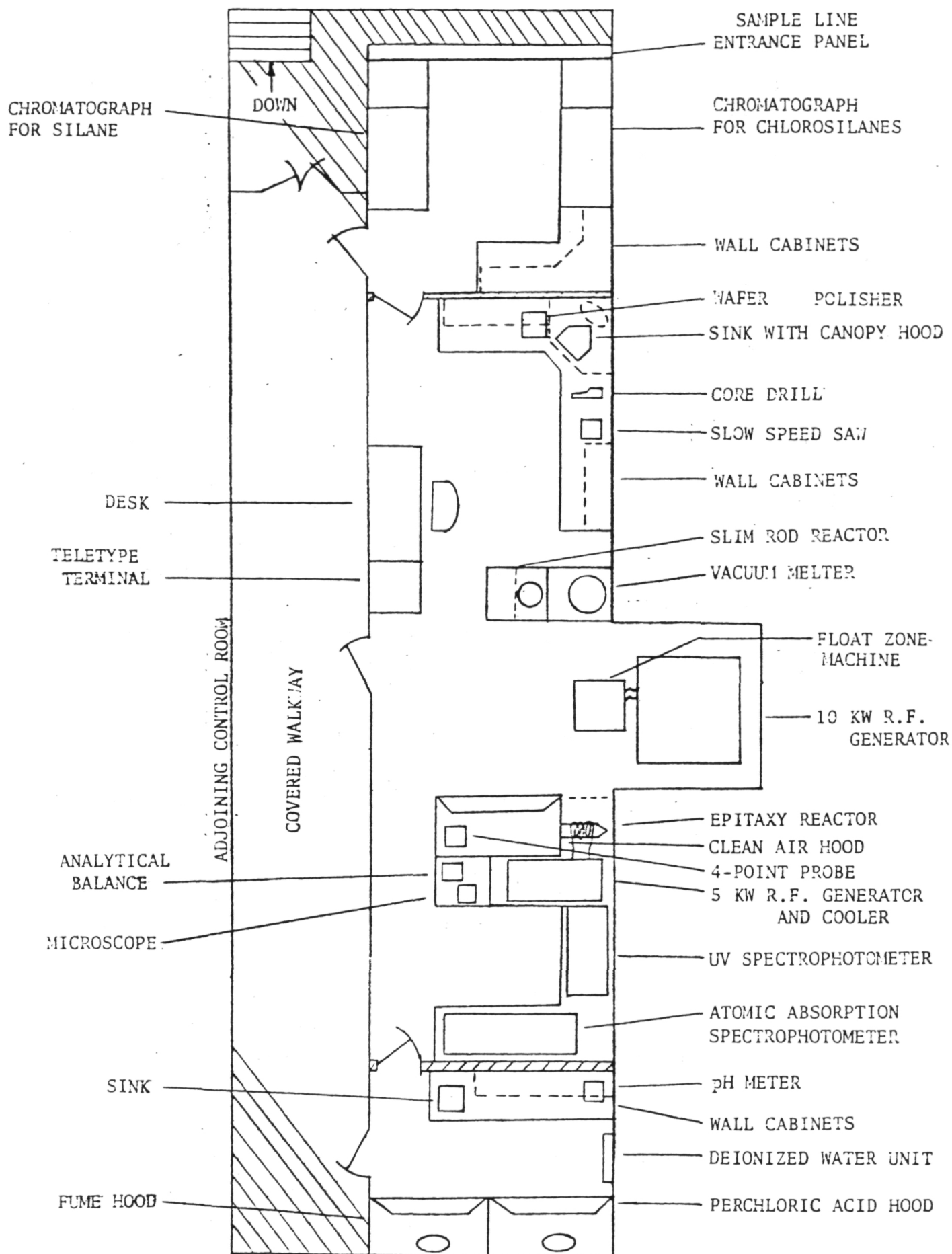
FIGURE 6.59 Proposed EPSDU Quality Control Laboratory

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of 4 to 10 μm per minute depending on the temperature and the reactant concentration. For example, the time required to deposit 90 percent new silicon is about 5 hours with a 2 mm diameter seed rod vs. 11 hours with a 5 mm diameter seed rod at a growth rate of 8 μm per minute. The resulting rods would be 6.3 and 15.8 mm diameter, respectively. Thus, the slim-rod reactor will convert to 2 mm diameter x 225 mm long seed rod into a 6.3 mm diameter polycrystalline rod ready for float zoning. The float zoning time is also diameter-dependent. It should take under 8 hours for a standard seven-pass operation of the 6.3 mm x 225 mm rod.

A fast and simple operation must be accomplished by purity maintenance throughout. All hot sections of the apparatus should be constructed of low vapor pressure, non-contaminating materials. The remaining sections should be made of materials having very low affinity for sorbing gases. Thus, the reactor shell, electrodes, and reactant supply piping will be made of stainless steel, water-cooled where necessary. The chucks, which hold the silicon seed rod to the electrodes, will be molybdenum which exhibits very low vapor pressure at the expected $1000^{\circ}C$ operating temperature, and also possesses good thermal and electrical conductivity.

1.6.4.1.2   Reactor Description and Preliminary Testing

The reactor components are arranged in three discrete modules (Figures 6.60 through 6.62):

- Power module - which consists of a 36 volt DC power supply and a ballasted 208 volt AC variable transformer.

- Control module - which consists of the analog recorder, infrared temperature controller, mass flow controller, meter relay, power control, and Gowmac thermal conductivity detector.

- Slim rod reactor - includes the main chamber, supporting structure, and gas and water plumbing. The silicon seed rod is supported vertically between heater-cooled electrodes contained within a water-cooled bell jar.
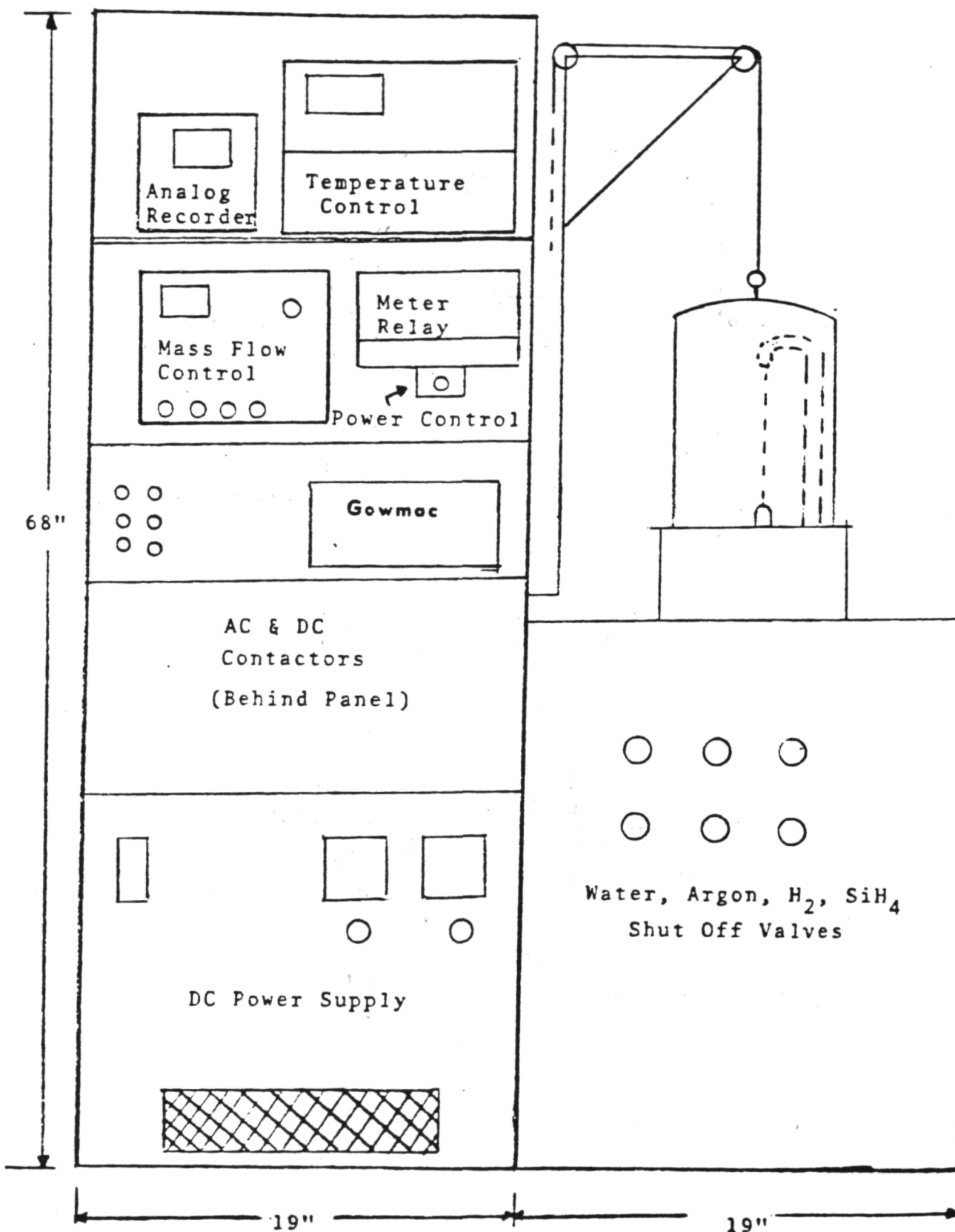
FIGURE 6.60   Slim-Rod Reactor System for Silane Quality Control

Bell Jar

Water-cooled
upper electrode

Molybdenum chuck

Slim Rod
of Seed Silicon

Tubing end connector

Silane entrance connection

Electrical Feed Through

Water-cooled lower electrode

Argon entrance connection
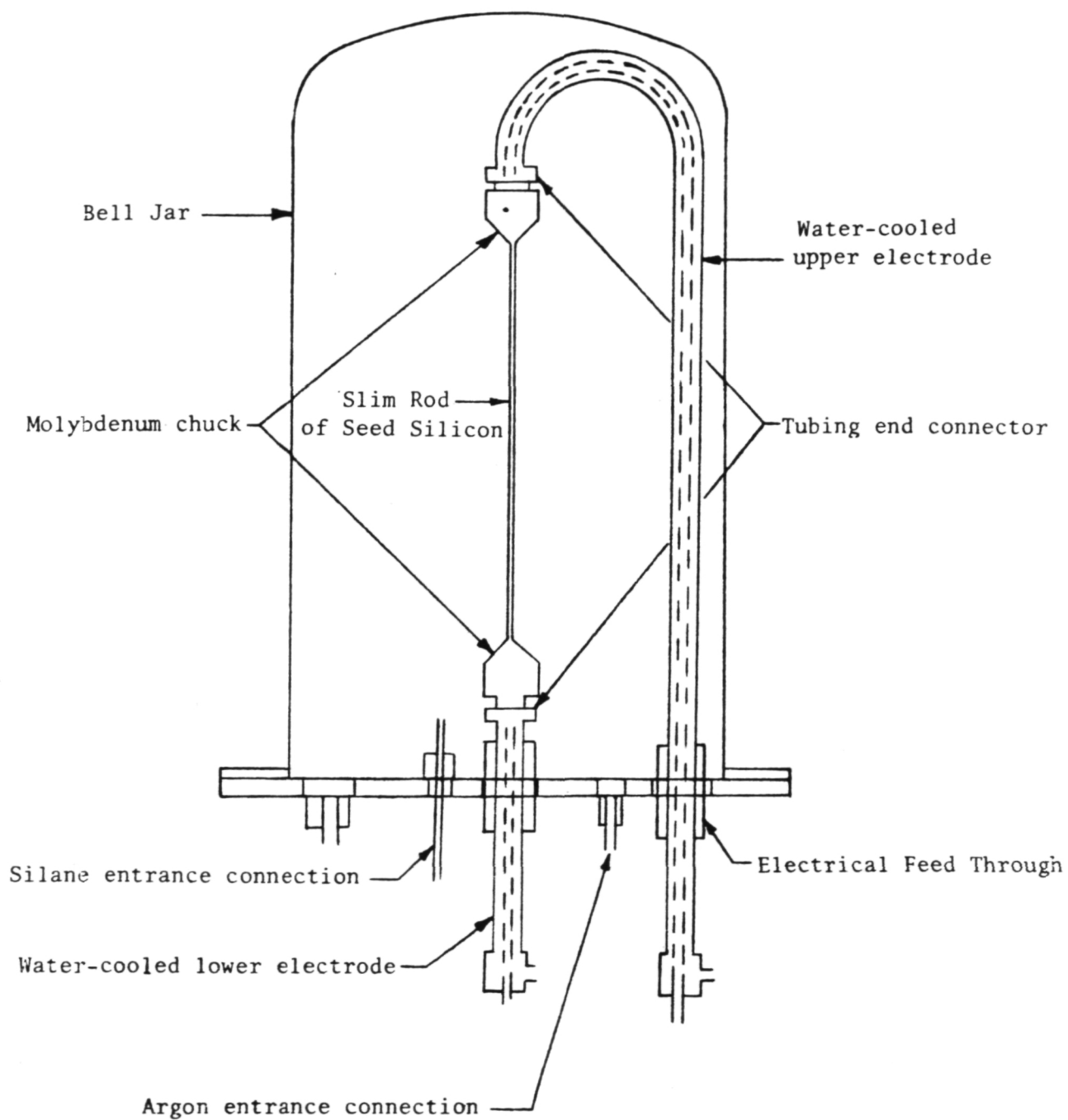
FIGURE 6.61   Slim-Rod Reactor for Silane Quality Control

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Eye Bolt for lifting

12 gauge 304 S.S. Plate

1" Nominal

1/4" Copper tubing
brazed to plate

Kovar/Glass
View Port

1-1/2" O.D. S.S.
Tube 1-1/2" long

12 gauge 304 S.S.
Plate rolled and welded

1/2" Conflat Flange

7.34"
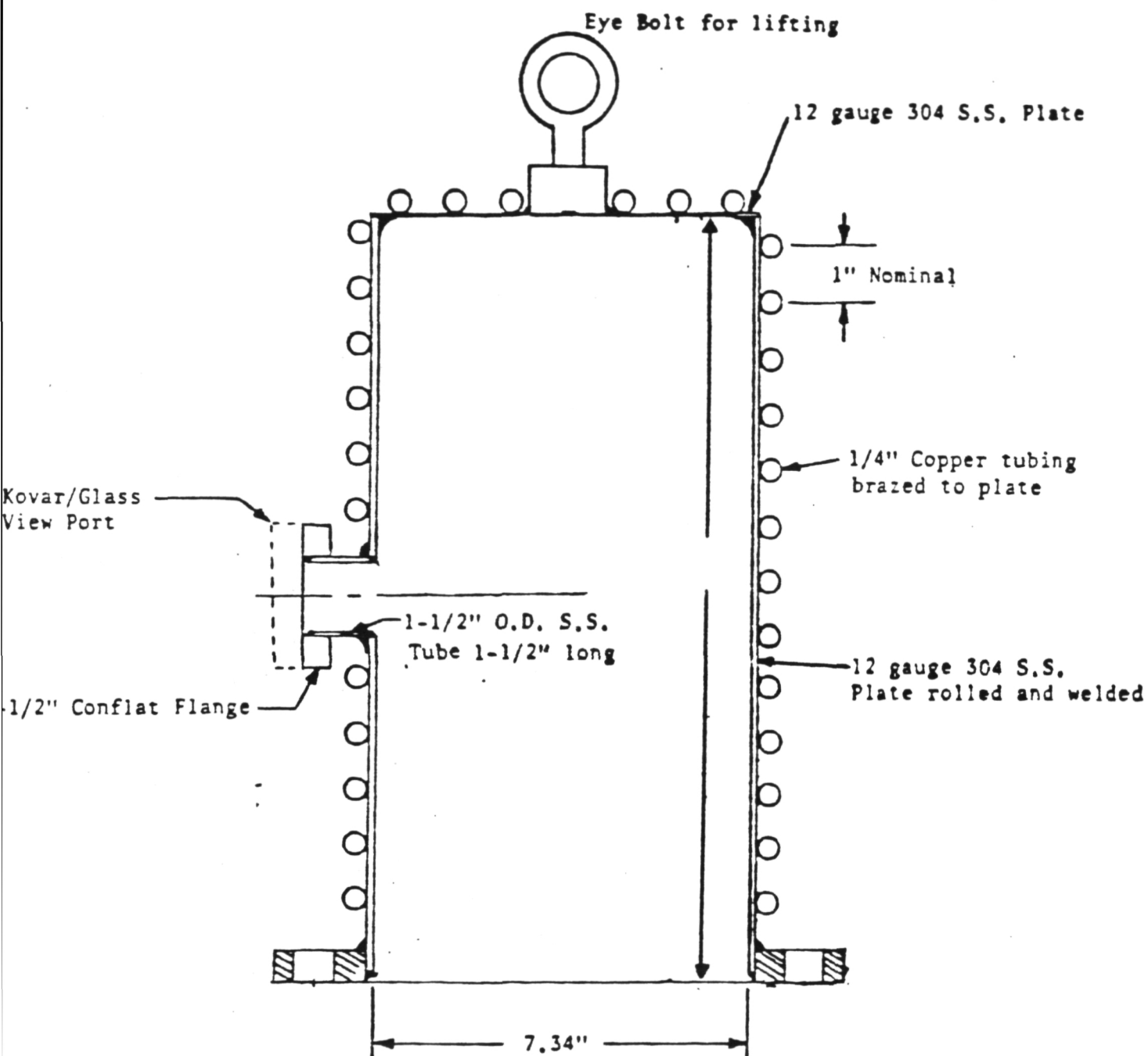
FIGURE 6.62   Bell-Jar - Slim Rod Reactor

The reactor shell is constructed of 12 gauge 304 SS and is wrapped with 1/4-inch copper tubing for water cooling. A Kovar glass view port is provided for the infrared temperature controller to sense and monitor the temperature of the silicon rod. The reactor internals consist of water-cooled upper and lower electrodes made of 304 SS, the molybdenum chuck to support the silicon rod, and the gas entrance connections. The reactor is designed to be operated from slightly above atmospheric pressure to a few millimeters of vacuum.

The addition of hydrogen, silane, and dopant (when used) gases are controlled by mass flow control valves. The gases are mixed in the gas delivery system before entering the reaction chamber. The silicon seed rod should be of modestly high purity to avoid gross contamination of the final rod, but yet have enough impurity to be conductive at voltages available in the laboratory. The resistance of silicon drops rapidly with increasing temperatures above $100^{o}C$. Therefore, the rod is preheated with the ballasted 208 volt AC variable transformer to overcome the resistance of the rod, thereby providing enough conduction through the high-purity silicon seed rod to heat it to the point where the low-voltage, high-current, DC power supply can operate. The silane gas entering the reactor is pyrolyzed on the seed rod depositing the silicon.

Several proving runs were conducted in the slim-rod reactor to define the rod operating temperature and the silane flow rate which yielded uniform silicon growth while minimizing amorphous silicon formation. Growth at $950^{o}C$ showed a smooth uniform surface texture, while at $1050^{o}C$ the growth was significantly more nodular. Brown amorphous silicon deposits were more pronounced at the higher temperature. At $950^{o}C$ rod temperature, the amorphous deposits do not appear to be a significant problem. At these rod temperatures, the bulk gas temperatures were always below $150^{o}C$ due to the large cooling effect of the reactor wall.

The silane flow was increased until brown powder formation was observed and then it was decreased slightly. At this point a trace of yellow color was observed in the burning off-gas indicating a small amount of un-reacted silane in the effluent. At $950^{o}C$ the silane flowrate was 15 cc/min;

at 1050°C the rate was 10 cc/min. The composition of the effluent was not measured because the thermal conductivity cell was not operational.

Several problems were identified during these proving runs. A substantial amount of silica found around the observation port was traced to a small leak in the flange gasket. Poor and erratic ohmic contact between the molybdenum chucks and the water-cooled electrodes resulted in short-term voltage spikes which blew the instantaneous type fuses in the low-voltage DC power supply. These fuses have been replaced with a time-delay type, and better contact has been made at the chucks.

The unit was unable to heat high-purity silicon seed rods without substantial external preheating. The unit was designed to utilize 2 mm square by 140 mm long rods of nominally 60 Ω cm silicon. That type of seed should be heated by a 200-volt source. The available seed rods were unable to be heated by direct resistance heating unless the length was shortened to 89 mm and the impressed voltage was increased to 600 volts. Those rods had a measured resistivity in excess of 500 Ω cm. The power dissipation required for a "cold start" is about 3.2 watts. The "normal" seed rods planned for use and currently being prepared have a 60 Ω cm resistivity and a 3 mm cross section.

An auxiliary heater was used to heat the seed rods to about 900°C where the rod resistivity is low. The voltage drop across the rod was less than 3 volts initially but increased to about 90 volts within a few minutes. Again, high-contact resistance is suspected. In addition, the rod bowed significantly because the slots in the molybdenum chucks failed to allow any linear movement of the expanding silicon filament. To resolve these deficiencies, high-purity graphite chucks will be prepared. These provided low contact resistance and were soft enough to allow for expansion of the silicon seed.

1.6.4.1.3   Summary of Tests

Polycrystalline silicon rods were routinely grown in the laboratory slim-rod deposition reactor. Using undoped, pure silane feed gas depositing

onto a 3 mm square x 150 mm long seed rod, a polycrystal rod of 8.25 mm square was grown in 14 hours. The average deposition rate in successive runs was nominally 1.0 gram/hour at $850°C$ deposition temperature. The bulk resistivity of the deposit was 253 Ω cm with a variation of 16 Ω cm along the rod as measured with a colinear four-point probe. Surface quality of the deposit was good. The growth was uniform over the length of the rod with a rounding off at the ends. No growth of silicon was evident on the water-cooled carbon chucks and, as shown in Figure 6.63, the end of the original substrate could be removed intact from the chuck. This provides a convenient, concentric mounting point for subsequent zone refining.

To complete the sequence as a quality control analytical method, the polycrystal rods were subjected to multi-pass zone refining and the resistivity profile measured along the resulting crystal. The fresh silicon deposit was intimately mixed with the seed during zone melting. The ratio of fresh silicon to seed was greater than 5.1. The quality of the first three polyrods grown defined the baseline silane quality.

During the growth, runs were not performed as the major activity but were to identify and resolve sources of contamination in the reagent gas supply system, portions of which are common to both slim-rod and epitaxy reactors. We previously established all parameters for routine uniform growth of silicon on heated seed rods from silane and further runs at this point, while adding to our experience factor, would have diluted our efforts toward resolving the silane purity problems.

The final experimental work using the slim-rod pyrolysis reactor was completed. A series of runs was made using silane having controlled concentrations of diborane or phosphine after a series of three runs using undoped silane were made to establish baseline quality. The objective of this program was to develop a technique which could be used to convert a sample of silane into a desirable form of silicon of equivalent quality which could be analyzed by standard techniques used by the industry; namely multi-pass float zone refining and resistivity profiling.

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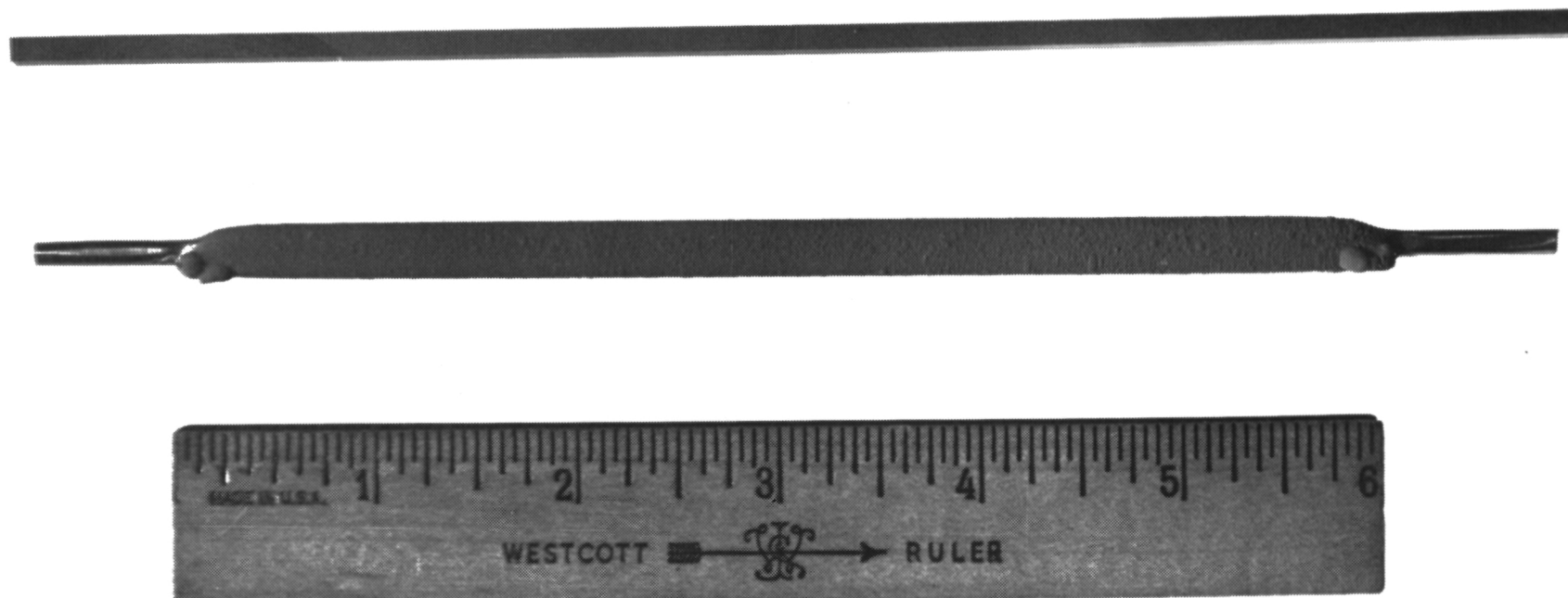
FIGURE 6.63   Slim-Rod Silicon Deposition

Three 14-hour growth cycles were made using high-purity silane gas. This gas had previously been used in the epitaxial reactor where film resistivities of $>150\,\Omega$ cm N-type had been measured indicating that the source gas was of high quality. The polysilicon growth onto the 3 mm square float-zoned single-crystal seed rods was smooth and uniform. The average rod diameter was 8.25 mm; the new silicon constituted 87% of the resulting rod. The resistivity of the rod, measured using a 4-point probe, was $250\,\Omega$ cm. This indicated that no gross impurities were present although more accuracy would require conversion of the polycrystal silicon rod to single crystal.

Three growth cycles were made with phosphine and three with diborane dopant gas mixed into the silane at various levels from 2 to 20 ppb. The dopant was injected into the silane after diluting the 50 ppm source gas with H_2. Again, 14-hour growth cycles yielded 8.8 mm diameter rods containing 87% new silicon.

1.6.4.2 Silane Analysis – Direct Measurement

1.6.4.2.1 Hydrocarbon Analysis

The development of a method for quantitative analysis of silane for parts-per-billion levels of methane, ethane and ethylene was completed. The use of an Ascarite (powdered sodium hydroxide) column to block passage of silane followed by a silica gel column to separate the hydrocarbons has been used to chromatographically analyze silane for methane, ethane, and ethylene. The limit of detection for methane is estimated at less than 100 parts-per-billion. The trace level analysis of silane for hydrocarbons will be useful in assessing the performance of the gas stripping column in EPSDU as well as providing a means of monitoring the performance of the final silane distillation column.

1.6.4.2.2 Phosphine Analysis

The determination of trace levels of phosphine in silane can be achieved with a detection limit of about 6 parts-per-billion (ppb) utilizing a gas chromatograph with a photoionization detector. This method should be utilized to augment other analytical techniques for silane quality control

in the planned 100 MT/Yr silane-silicon EPSDU.   The detection of low levels
of diborane by chromatographic separation from silane was not successful.

The Union Carbide Corporation process utilized the high vola-
tility of silane advantageously as a means for removing harmful impurities
through conventional distillation.   One of the closest boiling impurities
potentially present in silane (boiling point -112°C) is phosphine (boiling
point -87.4°C).   Phosphorus is an extremely potent donor-type impurity in
silicon and is not easily removed by normal unidirectional freezing methods
during crystal growth (segregation coefficient = 0.35).   Rapid, high-sensi-
tivity analysis of the silane intermediate provides important advantages for
control of silicon quality and trouble-shooting for trace contaminants.

Measuring the level of phosphine in silane in the low part-per-
billion range is important for assessing the performance of the final silane
distillation column, correlating silane quality with final silicon quality,
or providing a means for on-line doping to achieve consistent quality silicon.

The determination of trace quantities of electrically-active
impurities in silane and chlorosilanes has been historically performed by
deduction from the resistivity value of silicon films produced by decomposi-
tion of the silane in question.   Other methods, such as substoichiometric
separation - isotope dilution mass spectroscopy, low temperature infrared
spectroscopy, and wet chemistry methods have been proposed, but all require
the use of expensive equipment, time-consuming standardization, or preconcen-
tration steps.   The purpose of this present work was to develop an analytical
technique which was rapid, quantitative, and capable of ready application to
on-line quality control of silane purity.

A chromatographic technique was developed which can determine
the level of phosphine in silane at a detection limit of about 3 parts-per-
billion.   The heart of the technique is the use of a photoionization detector
which is particularly sensitive to phosphine.   A GOW-MAC 550 chromatograph
was equipped with an NHu, 10.2 electron volt photoionization detector operating
at 120°C.   The separation column was 6 feet of 1/8-inch O.D. stainless steel
tubing packed with Poropak P, 60-80 mesh.   At an oven temperature of 50°C

and 24 cc/min helium carrier flow, a 0.5 cc sample of 13 parts phosphine in helium was eluted in 1.46 minutes (Figure 6.64). The total area of 829,679 corresponds to a response of 8.42 x 10^{13} area counts/gram of phospine. Elution time and volume were reproducible.

A gas blend of 1 ppm phosphine in silane was prepared. The chromatograph of this sample, Figure 6.64, clearly shows the silane elution at 0.92 minute followed by the phosphine peak at 1.42 minutes. In this case, the integration calculation utilized a "tangent skim" method since the large silane volume has not been completely eluted. Using the same sensitivity factor determined for the phosphine-only case, the calculated quantity of phosphine was determined to be 1.142 ppm. Thus, it was confirmed that in the presence of silane, phosphine in trace levels can be separately eluted, that the sensitivity of the photoionization detector to phosphine is unaffected by the presence of silane, and that the "tangent skim" method of peak area determination is satisfactory.

For part-per-billion levels of phosphine, the sample size could be increased to 1.0 cc. Samples larger than 1-2 cc would not be as effective, because they would tend to overload the column and wash out the sensitivity. Assuming a 1.0 cc sample size and an ultimate sensitivity of 200 area counts for the "tangent skim", the limits of detection of phosphine would be 3 ppb.

1.6.4.2.3 Boron Analysis

Chromatographic determination of low levels of diborane in silane was not successful. To establish the type of chromatographic column which would quantitatively elute diborane and to establish detector sensitivity, gas mixtures containing 1% diborane in helium and 1% diborane in silane were prepared. Since diborane is extremely reactive with moisture, care was taken to thoroughly dry the helium carrier gas, column packing, sampling valve, and associated plumbing. The first problem encountered and solved was that a simple, hot-wire type thermal conductivity detector exhibited a non-reproducible response to diborane. Detector sensitivity varied with the sample

Chromatogram of 13 ppm PH$_3$ in He

Injection

1.46 (Phosphine)

Stop

TIME	TYPE	AREA	ID #	AMT
1.46	1	834316	1	13.13 PPM
			TOTAL	13.13 PPM

Injection

Chromatogram of 1 ppm PH$_3$ in Silane

0.60

0.92 (Silane)

1.42 (Phosphine)
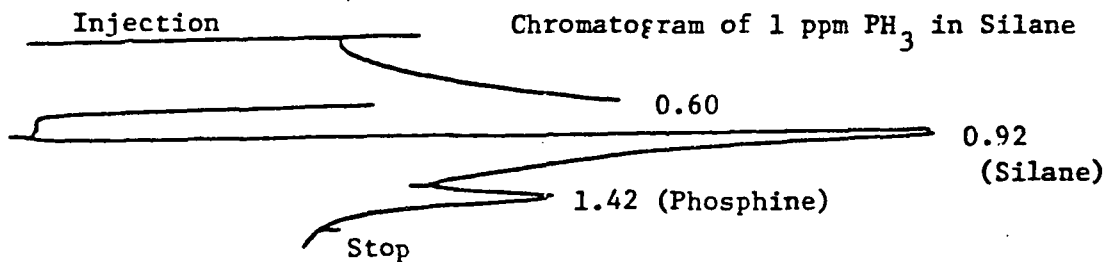
Stop

TIME	TYPE	AREA	ID #	AMT
.60		489151		
.92	IM	1827126		
1.42	T	72567	1	1.142 PPM
			TOTAL	1.142 PPM

Equip:	Gow Mac – 550
Carrier:	He at 24 cc/min.
Column:	6.0-ft. x 1/8-inch OD, S.S. Poropak P, 60-80 mesh
Oven Temp:	50°
Sample:	0.5 cc
Detector:	HNu 10.2 ev Photoionization type

FIGURE 6.64   Trace Phosphine Detection
by Chromatographic Method

injection frequency. A switch to a thermistor-type detector, which operates at a much lower temperature, appeared to solve that problem so that a reproducible detector response was measured for a given quantity of diborane injected. With a sensitivity of 500 area counts/10^{-7} gm of diborane on the GOW-MAC 500/HP 3388 unit used, this provided an ultimate detection limit of 50 ppm.

Several column packings were evaluated first to achieve a delayed (with respect to the carrier gas flow), yet quantitative elution of diborane, and then later to achieve a differential elution volume between diborane an silane. Since diborane is thermally unstable, a low temperature control system was provided to evaluate chromatographic separation at temperatures as low as -40°C.

COLUMN PACKING EVALUATION FOR DIBORANE

Mixture

Packing	Diborane/Helium	Diborane/Silane
Carbopack	No elution	-
Silicon Oil	No elution	-
Dioctylphthalate	OK	No separation
Poropak QS	No elution	-
n-hexadecane	OK	No separation

All tests were performed over a temperature range of -40°C to $+40^{\circ}$C.

Since these column packings cover a broad spectrum of polarity and activity, the chance of finding a suitable system for achieving good separation did not appear promising. Reactive techniques similar to those applied to the determination of hydrocarbons in silane do not work well with diborane since diborane, rather than the silane, is the more reactive with most common reagents. Further, even if a chromatographic separation could be achieved, the modest detector sensitivity would give a lower limit of detection of only

about 50 ppm. Other detectors, such as flame photometric or photoionization, are not very sensitive to boron as compared to the excellent results which can be obtained with phosphorus compounds.

Further analysis revealed that diborane's rapid hydrolysis in water, as compared with silane, offers a possible wet chemical technique whereby a relatively large volume of silane could be bubbled through acidified water. The acidified water could then be analyzed for boron using standard methods of complexing with curcumin or carminic acid and measuring its UV absorbance.

With the poor detector response to diborane and the failure to achieve a significant chromatographic separation between diborane and silane, work in this area was suspended.

1.6.4.2.4   Diborane/Silane Vapor - Liquid Equilibria

The vapor-liquid equilibria (VLE) of a diborane/silane mixture was measured at $40^{o}C$ and 300 psi, the planned operating point of the EPSDU silane distillation column. The relative volatility of silane over diborane was measured as 2.03 $\pm$ 0.07 at a diborane concentration of one percent. This helps to substantiate the operating basis of the EPSDU silane column which is designed to separate the more volatile silane in a high tray-count column. Tests for thermodynamic consistency and confirmation of the VLE model used for the SiH_4/B_2H_6 system is planned.

The experimental technique utilized in this determination involved condensing a mixture of silane containing one percent diborane into a liquid-nitrogen-chilled equilibrium cell. The cell was cooled to $-40^{o}C$ where it was maintained with mechanical agitation to assure equilibrium conditions. Through a plumbing system submerged in the $-40^{o}C$ bath, samples of vapor and liquid were withdrawn. The samples were analyzed by passing the vapor (or vaporized liquid) through a small quantity of high purity water containing a pinch of curcumin. The amount of boron trapped was measured photometrically using a UV spectrophotometer. Several determinations were made on different days with

good agreement using them. An evaluation for thermodynamic consistency shows the system to be nearly ideal.

The original EPSDU column design was made assuming ideal gas rules, and hence the design basis has been confirmed; no further modeling or changes in column design are required.

1.6.4.3 Silane Epitaxy

The purpose of this work was to develop a rapid analytical technique for qualitatively determining the electronic quality of silane produced by EPSDU prior to committing the silane to the decomposition reactor. The technique is based on forming an epitaxial film of silicon from the silane sample onto an N or P-type single crystal substrate wafer and measuring the film resistivity. Net donor or acceptor levels can be inferred and a minimum resistivity specification can be established for silane suitable for conversion into product silicon.

1.6.4.3.1 Reactor Design

A design (Figure 6.65) has been prepared for a reactor system for controlled epitaxial deposition of silicon from silane onto single crystal wafers. The reactor system consists of a reactant gas metering system, an inductively heated quartz reactor, and a vacuum and vent system. The gas metering system is identical with that constructed for the slim-rod reactor including provision for on-line double dilution doping of the silane reactant. Solenoid valves in the gas supply lines permit system automation for routine reproducible analytical work. The reactor itself consisted of a quartz tube with surrounding RF induction coil. A quartz boat holding a carbon susceptor at a slight angle to the flowing gas held two or four silicon wafers of 1 to 2-inch diameter. A special entrance arrangement permitted introducing the reactant gases, optical temperature sensing, and a means for inserting the silicon wafers. This end of the reactor was housed inside a filtered air hood to provide a dust-free environment. Air cooling of the quartz tube was provided to minimize silane decomposition on the tube walls. The vacuum system was provided for purging and degassing the system between runs to eliminate absorbed impurities from causing spurious results.
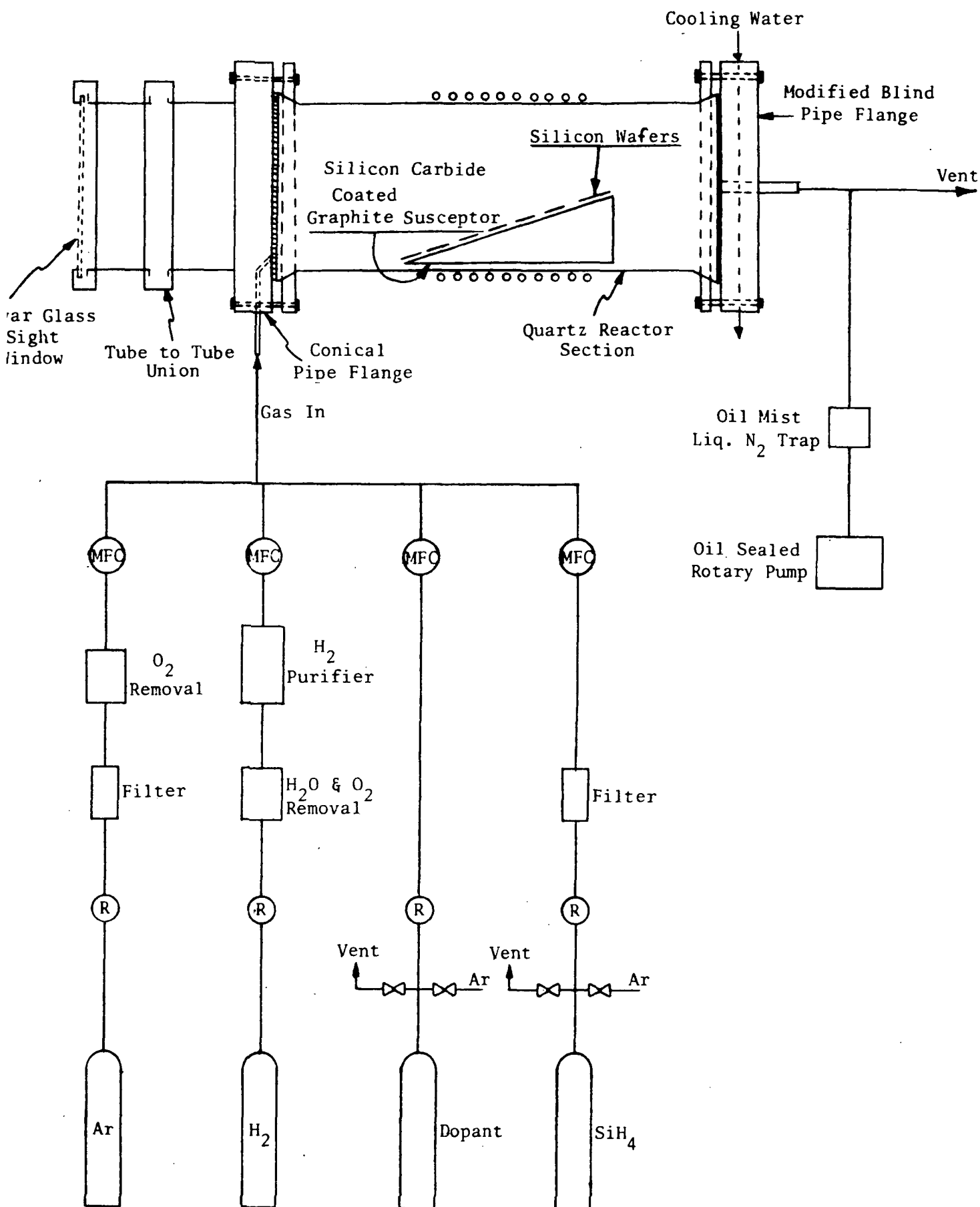
FIGURE 6.65   Epitaxy Reactor

The figure shows an epitaxy reactor with the following labeled components:

- Cooling Water
- Modified Blind Pipe Flange
- Vent
- Silicon Wafers
- Silicon Carbide Coated Graphite Susceptor
- Quartz Reactor Section
- ar Glass Sight Window
- Tube to Tube Union
- Conical Pipe Flange
- Gas In
- Oil Mist Liq. N_2 Trap
- Oil Sealed Rotary Pump
- MFC
- O_2 Removal
- H_2 Purifier
- Filter
- H_2O & O_2 Removal
- R
- Vent   Ar
- Vent   Ar
- Ar
- H_2
- Dopant
- SiH_4

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1.6.4.3.2   Reactor Testing

The air-cooled epitaxy reactor was operated to produce nominally 15 μm films on 30 mm diameter, 60 Ω cm P-type wafers from undoped silane. Wafer cleaning techniques were defined to substantially eliminate crystal imperfections. Several sources of contamination were identified and corrected including a "dirty" HCl regulator, and a H_2 purifier element.

Epi-film thickness was measured non-destructively using differential interference contrast microscopy to measure stacking fault dimensions which are directly related to film thickness as taught by ASTM 143-73. A film thickness calculated from wafer weight gain was compared with the optically determined value as a potentially quick method of thickness determination. To provide sufficient stacking faults, each fresh wafer was probed with the four-point probe to inflict modest surface damage prior to epitaxy growth.

The four-point probe utilized for measurement of film resistivity was checked using a fresh wafer with an independent four-point probe and found to read within one percent. All resistivity measurements on epitaxy films are made with the specimen located in a light-tight box to avoid photovoltaic effects. An adjustment was provided for compensating for epi-film thickness. Conductivity type was determined by the rectifying technique for bulk specimens and by the thermoelectric method for the epi-film.

In determining the "base line" resistivity of the undoped silane supply, multiple runs were made in a single day using, as near as possible, identical wafer handling and reactor operational techniques. After cleaning the fresh wafer in an ultrasonic detergent bath, rinsing with electronic-grade methanol and Type I water and clean-air drying, the specimen is loaded onto a silicon-coated graphite susceptor and placed into the continuously purged epi-reactor tube. An H_2 purge while heating to $1100^{\circ}C$ is followed by a 10-minute HCl etch and cooling to $1050^{\circ}C$. At that temperature a 20-minute deposition period with 0.5% SiH_4 in H_2 produces a 15 μm film. Electrical measurements are made on the cooled wafer.

A low average film resistivity with large run-to-run variation was observed when nominally 15 μm films were grown from a single source of silane on 60 Ω cm P-type wafers. Various trouble-shooting activities showed that improvement in resistivity values were obtained when:

- The ultrasonic wafer pre-cleaning bath was cleaned at prescribed intervals.

- Several fittings contaminated with C-100 thread lubricant, were identified and cleaned.

- The silane, hydrogen, and HCl supply lines were acid cleaned, rinsed, and dried.

A significant step increase from approximately 40 Ω cm to over 100 Ω cm was obtained when an alternate silane supply cylinder was used. Both cylinders were presumably "electronic grade" with a resistivity of >100 Ω cm when manufactured. The second cylinder had about 6 months shorter time prior to use. The reasons for low purity in the first cylinder were not established. The second cylinder was used in all subsequent work. Even after these sources of contamination, which resulted in higher average resistivity values, were identified and resolved, there was still a significant variation in the measured epi-film resistivity.

A series of runs were made varying the duration and concentration of the HCl etch. HCl is potentially the dirtiest of the gases used in the epitaxy test, thus, the concentration and duration of the pre-etch could have significant impact on final film quality. As listed in Table XVI, the surface quality of the film and the resistivity variation across the wafer improved when the HCl etch was reduced in concentration (flow rate) while etching for a slightly longer period. The H_2 carrier flow was constant at 6 liter/min, through the 38 mm ID reactor as was the etch temperature of 1125°C. An HCl concentration of 0.42% for 10 minutes produced wafers which were free of haze and substantially free of stacking faults.

TABLE XVI

EFFECT OF HCl ETCH ON EPITAXY FILM QUALITY

	HCl Etch									
Run #/ Page	Flow Rate cc/cmin	Meter Setting	Time Min.		Resistivity Ω cm					Wafer Appearance
1/82 (83A)	60	112	8	100	188 160 154.5	190	0.03289 g			slight cloudiness on front, Monsanto wafer
2/83 (83B)	60	112	8	248	229 223 157	277	0.03468 g			cloudy all over no striation, heavier on front
3/83 (83C)	60	112	8	236	540 250 203	182	0.03372 g			cloudy all over heavier at front striations visible
4/84 (84)	60	112	8	224	284 161 161	145	0.02994 g			cloudy all over heavier at front striations visible
1/85 (85A)	12	30	8	47	40 56 33	53	0.02139 g			cloudy at frony slightly stacking faults evident all ove
2/85 (85B)	12	30	5	45	62.9 54.3 47	48.8	0.02771 g			stacking faults all over
3/85 (85C)	60	112	5	38.6	75.4 75.7 70.9	77.4	0.02927 g			cloudy at front striations visible stacking vaults all over
4/86 (86A)	25	50	5	75.5	349 95 81.9	116.7	0.02666 g			very slight cloudiness at front stacking faults all over
5/86 (86A)	25	50	6	85.6	136.5 156.6 131.8	184.1	0.02468 g			very slight cloudiness cloudy spot at front few stacking faults

TABLE XVI (CONT'D)

Run #/ Page	Flow Rate cc/min	Meter Setting	Time Min.	Resistivity Ω cm			Wafer Appearance
1/87 (87A)	25	50	6	91.5	102.4 96.9 86.6	144.9 0.02737 g	slight cloudiness at front - some stacking faults
2/87 (87B)	25	50	6	157.8	120.4 107.2 84.7	135.7 0.02785 g	some cloudiness at front some stacking faults
3/87 (87C)	25	50	6	40.1	63.9 52.0 47.8	62.6 0.02661 g	deposition at 1000°C zone of thick cloudy deposition - remainder clear - few stacks
4/88 (88A)	25	50	10	156.6	184.6 157.0 124.9	164.6 0.02939 g Av 157.5	very very slight cloudiness at front stacking all over
5/88 (88D)	25	50	10	124.1	112.0 97.9 102.0	100.3 0.02901 g Av 107.3	wafer clean few stack
1/89 (89A)	25	50	10	439	242 399 783	401 0.02687 g Av 370	clean wafer
2/89 (89B)	25	50	10	158	175 121 140	197 0.02656 g Av 158.2	clean wafer - several stacks
3/89 (89C)	25	50	10	163	137 162 180	196 0.02398 g Av 167.6	some cloudiness left side - very few

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There were slight resistivity variations across the wafer surface which are largely due to variations in film thickness. However, when five measurements are made at the positions shown in Figure 6.66, the average values are within 4% of the maximum variation. Higher resistivity values and thinner deposits were obtained on the initial test on any given day. This could be due to inadequate silane purge to remove the overnight nitrogen purge gas. This fact is significant for EPSDU operations where purging between samples will be very important for assuring representative results.

Expitaxial films of consistently high resistivity can be grown using a well-defined regime of precleaning and high temperature processing.

The series of runs were made during December 1980 by adding controlled amounts of phosphine or diborane to silane. The purpose was to develop a correlation between impurity level in the sample silane and the resistivity of the deposited film.

For the case of phosphine, the deposits were made on P-type wafer substrates in order to form a N - P junction and permit measuring the epifilm resistivity. Figure 6.67 shows the result of those runs along with a plot of the theoretical resistivity assuming all of the phosphine had been incorporated.

A similar set of experiments was made using N-type wafer substrates. However, the baseline resistivity values using undoped silane (165 Ω cm, N-type) were erratic, varying considerably from edge to edge across the wafer and also from wafer to wafer. This is not too surprising since relatively high-purity N-type silane was used as the control and thus, no junction was being made. One dopant run, using 17 ppb B_2H_6, resulted in a wide distribution of resistivity readings.

A report detailing the epitaxy test method and results was written. In spite of the erratic results with the diborane dopant run, the general method is felt to be valid for determining silane quality quickly and sufficiently accurate to meet EPSDU's needs.
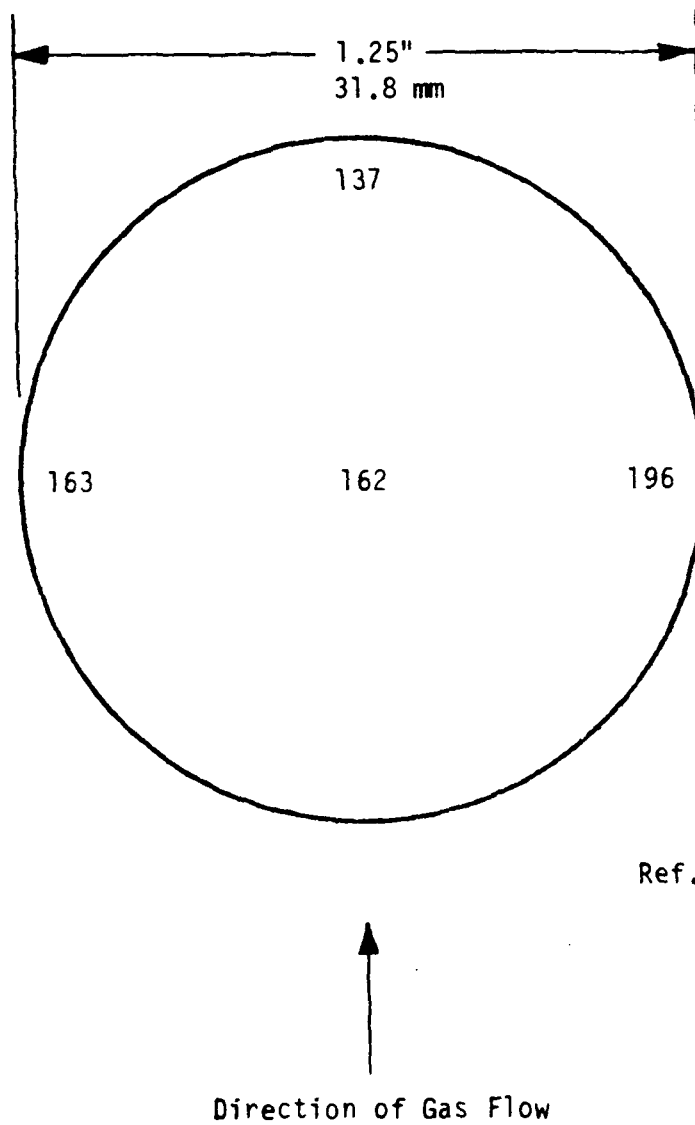
FIGURE 6.66   Resistivity Variation with Position
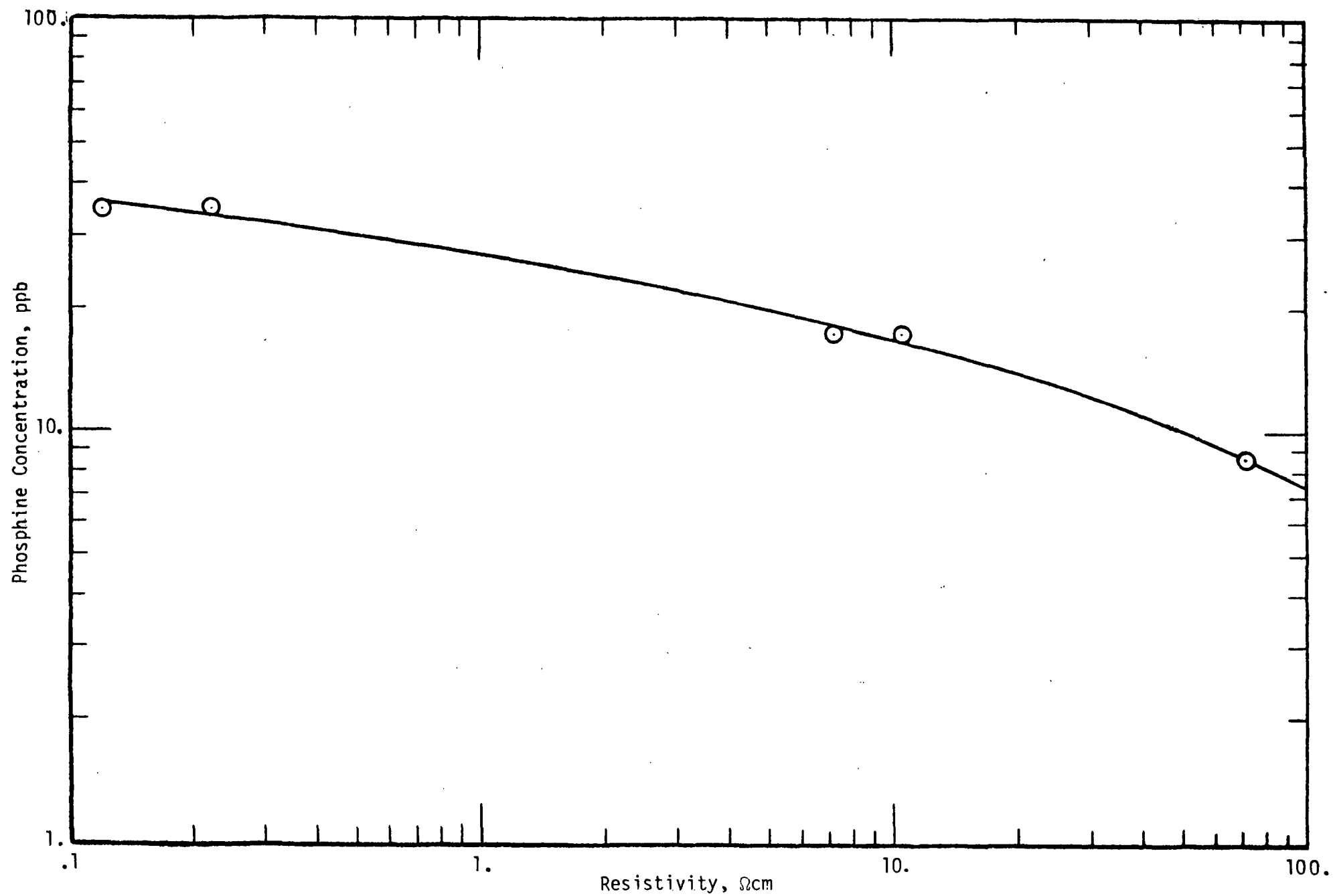
FIGURE 6.67   Resistivity vs Dopant Concentration - Silane Epitaxy on 60 Ωcm, P-type, Silicon Substrate

1.7 MANAGEMENT AND DELIVERABLES

Phase III of the contract began in April 1979 and continued through the fourth quarter of 1981. The Performance Measurement System (PMS) was instituted on January 1, 1980, to have a method to monitor and control the project and provide information and accounting to the contractor (JPL). The EPSDU program was divided into seven identifiable report items and further subdivided into tasks and work items.

The report items are as follows:

    1.1 Design/Procurement
    1.2 Equipment Fabrication
    1.3 Installation/Checkout
    1.4 Operation
    1.5 Commercial Process Economic Analysis
    1.6 Process Support R&D
    1.7 Management and Deliverables

Refer to Table I for the complete work breakdown structure (WBS).

During the program the following documents were delivered:

- Monthly Technical Progress Reports: This report gives the technical progress during the previous month, is written such that each work item is addressed.

- Monthly Financial and Management Reports: This document reports expenditures and estimated costs for the next three months and balance of contract. In addition, the detailed schedule is shown on a chart form and identifies major milestones accomplished or planned for each activity.

- Quarterly Technical Progress Reports: This report summarizes the previous quarterly progress, is published and widely circulated to companies with interests in the solar programs.

- Procurement Status Reports: Equipment purchase orders are identified and the delivery status is reported each quarter.

In addition to the monthly and quarterly reports, meetings were held to monitor progress.

- Bi-Monthly Reviews: The program is closely monitored by JPL and reviews are held every other month to assure that the program is on schedule and to discuss technical achievements or problem areas.

- Program Informational Meetings (PIM): These meetings are held about three times per year and are attended by participants in the solar programs. A slide presentation is usually made to discuss the program's status and future plans.

The program has been successfully accomplished through Design/ Procurement and Equipment Fabrication and partially through Installation. However, due to recisions of government funding, the program appeared to be in jeopardy until a new program was offered by UCC. This plan proposed that UCC would undertake to carry out the major objectives of the original plan with private funding.

Under UCC proposal the EPSDU facility will be relocated on the West Coast. The purchased equipment will be transferred from the East Chicago location, and the facility completed at UCC expense with a planned start-up in the third quarter of 1982. Since Phase III has terminated prematurely, this final report (Phase III) is written to collect and summarize the results during the period of April 1979 through December 1981.

Report Item 1.4, Operation, was limited to preparation, inspection of equipment and writing an operating manual. The manual is partially complete and will be a "deliverable" under the cooperative agreement.

Report Item 1.5, Commercial Process Economic Analysis, was completed to the point of developing a 1000 MT/Year commercial design, however, activity was curtailed in 1980.

Report Item 1.6, Process Support R&D, is continuing at a reduced level but in order to separate fiscal year 1982, costs will be assigned work item 1.8.

Report Item 1.7, Management and Deliverables, will be curtailed upon completion of documentation; however, Management/Reporting, etc., of the Fluid-Bed R&D program for fiscal year 1982 will be continued under a new Report Item, 1.9.

Milestones

Milestones were identified in the performance measurement system (PMS) to measure progress. The achievement of each major milestone was reported quarterly:

a. 1st Quarter 1980

WBS No.	Milestone	Description of Major Milestone
1.1.3.2	(A)	Electrical one-line diagram issued.
	(D)	Electrical Power System equipment specifications complete, RFR's issued.
1.1.5.1	(A)	EPSDU Facility Cost estimate issued.
1.6.1.1.3	(B)	First silane decomposed in Free-Space Reactor (FSR) - powder sample available for shipment.
1.6.1.1.4	(C)	Run No. 1 in FSR complete - internal report issued.
1.6.1.2.2	(F)	Internal report issued on development of new FSR model.
1.6.2.1.2	(B)	Sub-contract signed for development of melter.
1.6.3.1.3	(A)	Internal report issued on Fixed-Bed Deposition.
1.6.3.3	(C)	Internal report issued evaluating silicon deposition experiments in a fixed-bed reactor.

WBS No.	Milestone	Description of Major Milestone
1.6.4.2.2	(M)	Draft report completed on Chromatographic Methods for Boron & Phosphorus Determination.
1.6.4.3.1	(B)	Epitaxy reactor final process flow sheet and equipment purchase orders issued.
1.6.4.3.2	(E)	Epitaxy reactor in place and ready for system connections.
1.7.3	(B)	Hydrogenation Reactor Design memo issued.
	(I)	Chromatographic Methods for Boron & Phosphorus Determination memorandum issued.

b. **2nd Quarter 1980**

WBS No.	Milestone	Description of Major Milestone
1.1.1.1	(B)	Final process design package issued.
1.1.1.3	(A)	Waste burner functional specification issued.
1.1.2.1	(A)	Final facility layout complete, drawings released.
1.1.3.3	(G)	UCC-designed process equipment specifications complete, RFR's issued.
1.1.3.4	(B)	Vendor-designed process equipment specifications complete.
1.1.3.5	(C)	Auxiliary equipment specifications issued.
	(E)	Auxiliary equipment RFR's issued.
1.1.3.6	(F)	Support equipment specifications issued.
1.1.4.1	(A)	Site preparation package issued for bids.
	(B)	Site preparation subcontract awarded.
1.5.1.1	(A)	Commercial process functional package issued.
1.6.1.1.4	(D)	Run Nos. 2 and 3 in FSR complete - internal report issued.

WBS No.	Milestone	Description of Major Milestone
1.6.1.1.5	(E)	Internal report FSR experimental results issued.
1.6.3.2.2	(B)	Internal report issued documenting results of particle separation tests in a fluidized silicon bed.
1.6.3.4.1	(D)	Fluid Bed reactor drawings released for fabrication.
1.6.4.1.3	(C)	Slim-rod reactor checkout complete - silane introduced into reactor.
1.6.4.1.4	(D)	Slim-rod reactor startup complete - first silicon deposited on seed rod.
1.6.4.3.3	(F)	Epitaxy reactor installed and checked out - operational safety review completed and operating procedures approved.
1.7.3	(C)	Silane Column Design memo issued.
	(E)	Definitive EPSDU Cost Estimate issued.
	(G)	Free Space Reactor Theoretical Analysis report issued.
	(H)	Evaluation, Fixed Bed/Fluidization memo issued.

c. 3rd Quarter 1980

WBS No.	Milestone	Description of Major Milestone
1.1.2.2	(B)	Safety checklist and environmental design report issued and permit obtained.
1.1.4.2	(C)	Civil/Structural package issued for bids.
1.1.4.2	(N)	Civil/Structural subcontract awarded.
1.1.4.3	(D)	Civil/Structural drawings issued for bid package.
1.3.1.1	(C)	Site work completed.
1.3.1.2	(B)	Civil work started.

WBS No.	Milestone	Description of Major Milestone
1.6.1.1	(G)	Modification to the free-space reactor PDU and hopper completed.
1.6.4.3	(G)	EPI reactor startup completed and first silicon deposition made on silicon.
1.7.3	(D)	Report EPSDU waste burner design issued.
	(F)	Report Free Space PDU experiment report issued (covering work thru June 1980).
	(J)	EPSDU equipment specification package issued.

d. 4th Quarter 1980

WBS No.	Milestone	Description of Major Milestone
1.1.2.3	(C)	Scale model complete.
1.1.3.8	(K)	Data Collection System - Equipment RFR's issued.
1.3.1.3	(D)	Structural work started.
1.6.1.1.7	(H)	Purity & operability tests - consecutive runs and 12-hour duration runs complete.
1.6.4.1.4	(H,K)	Method Development - Dopant Test Series No. 1 complete.
1.6.4.3.4	(L)	Method Development - Silane Analysis - Expitaxy report draft issued.

e. 1st Quarter 1981

WBS No.	Milestone	Description of Major Milestone
1.1.3.1	(K)	P&I diagram issued and control panel RFR's issued in March.
1.3.1.2	(F)	Complete civil work.
1.3.1.3	(J)	Completed structural work.
1.7.3	(K)	Engineering Design Package
	(L)	Installation Drawing Package

WBS No.	Milestone	Description of Major Milestone
	(M)	Interim Report on Subcontract
	(N)	Slim Rod Reactor Report
	(P)	Silane Analysis - Epitaxy

f. 2nd Quarter 1981

WBS No.	Milestone	Description of Major Milestone
1.1.1.2	(C)	Installation design review complete. Meeting minutes issued.
1.1.1.4	(A)	Process design package issued.
1.1.4.4	(E)	Mechanical/piping package issued for bids.
1.1.4.5	(F)	Drawings issued for bid package.
1.1.4.6	(G)	Electrical package issued for bids.
1.1.4.7	(H)	Electrical drawings issued for bid package.
1.1.6.1	(O)	Control system design P&I package started.
1.4.1.1	(O)	Operating manual started.
1.6.3.4.2	(E)	PDU system checkout completed.
	(Q)	"Arc discharge" heating of silicon bed completed.
1.2.8.2	(E)	Equipment inspection report issued.
	(F)	Specialty items delivered to site.

g. 3rd Quarter 1981

WBS No.	Milestone	Description of Major Milestone
1.1.2.6	(A)	Gantry scale model update - complete.
1.2.8.3	(G)	Vendor print control - all drawings on file.
1.4.1.3	(O)	Test plan started.

2.0   REFERENCES

1.   Eversteijn, F. C. and Put, D. M., J. Electrochem. Soc.:
        Solid State Science and Technology, 120, 103 (1973).

2.   Murthy, T. V. M. S., et al, J. Crystal Growth 33, 1 (1976).

3.   Eversteijn, F. C., Phillips Res. Repts. 25, 135 (1971).

4.   Timmel, P. J. and Rexer, J., Silane Pyrolysis in a Fluid-Bed
        Reactor, Report No. PR-79-230.

5.   Osada, Y., et al, J. Electrochem. Soc: Electrochem. Science
        and Technology 126, 31 (1979).

3.0   GLOSSARY

This section includes symbols, notations and abbreviations used in this report.

NOTATION	DESCRIPTION
A/D	Analog to Digital
Btu	British Thermal Unit
cm	Centimeter
C	Celsius
CRT	Cathode Ray Tube
DCS	Dichlorosilane
EPSDU	Experimental Process System Development Unit
F	Fahrenheit
ft	Feet
FBR	Fluid-Bed Reactor
FSR	Free-Space Reactor
g	Gram
hr	Hour
hp	Horsepower
HCl	Hydrogen Chloride
I/O	Input/Output
I.D.	Inside Diameter
JPL	Jet Propulsion Laboratory
kg	Kilogram
KV	Kilovolts
KW	Kilowatt
Kpa	Kilopascals
Khz	Kilohertz
lb	Pounds
LSA	Low-Cost Solar Array
m	Meter
MGS	Metallurgical Grade Silicon
mm	Millimeter
MM	Million
MT	Metric Ton
min	Minute

GLOSSARY OF TERMS (continued)

NOTATION	DESCRIPTION
MIT	Massachusetts Institute of Technology
MCS	Monochlorosilane
MCC	Motor Control Center
O.D.	Outside Diameter
ppb	Parts per Billion
ppm	Parts per Million
psi	Pounds per Square inch
psia	Pounds per Square inch Absolute
psig	Pounds per Square inch Gage
P	Pressure (atmospheres)
P.O.	Purchase Order
PSR	Procurement Status Report
PMS	Performance Measurement System
PDU	Process Development Unit
P&I	Process and Instrumentation
Q	Heat Flux
Q.C.	Quality Control
R&D	Research and Development
RFR	Request for Requisition
RFQ	Request for Quotation
sec	Second
SEM	Scanning Electron Microscope
SMS	Shotter Melter System
STC	Silicon Tetrachloride
SCFM	Standard Cubic Feet per Minute
TCS	Trichlorosilane
U	Superficial Gas Velocity (m/sec)
U_{mf}	Minimum Fluidized Velocity (m/sec)
UCC	Union Carbide Corporation
yr	Year
μm	Microns
Ωcm	Resistivity, OHM-Centimeter
Δp	Pressure Difference

NOTATION	DESCRIPTION
ρ_b	Bulk Density of Silicon
ρ_s	Silicon Particle Density
ϵ	Void Fraction

APPENDIX A

LIST OF MAJOR EQUIPMENT ITEMS

SILICON PROJECT
EQUIPMENT LIST

TAG NUMBER	DESCRIPTION	BM/SPEC NUMBER		QUANTITY ORDERED	P. O. NUMBER
01-02	Mobile Laborat	ES-7214	Rev 2	001	50096-01
11-02	Storage Bin	ES-6857	Rev 2	001	50033-01
17-02	Dust Bag	ES-7594		001	50187-01
21-02	Hopper Tank	ES-6852	Rev 3	001	50035-01
21-04	Settler Tank	ES-6852	Rev 3	001	50035-02
21-06	Chloride Tank	ES-6852	Rev 3	001	50035-03
21-08	Cond Receiver	ES-6852	Rev 3	001	50035-04
21-10	Storage Tank	ES-6852	Rev 3	001	50035-05
21-12	Storage Tank	ES-7278		001	50017-01
21-14	H_2 Receiver	ES-6852	Rev 3	001	50035-06
21-16	H_2 Humid Tank	ES-7278		001	50017-02
21-18	Blender/Tank	ES-6852	Rev 3	001	50035-07
23-02	H_2 Compressor	ES-6725	Rev 2	001	50010-01
23-03	H_2 Compressor	ES-6725	Rev 2	001	50010-01
24-02	Quench Condenser	ES-6801	Rev 3	001	50014-01
24-04	Vaporizer	ES-6798	Rev 3	001	50015-01
25-02	Hydrogenation	ES-6835	Rev 2	001	50056-01
26-02	LMV-801 Pump	ES-6718	Rev 2	001	50005-01
26-04	Diaphragm Pump	ES-6720	Rev 2	001	50245-01
26-06	STC Pump	ES-7193	Rev 1	001	50105-01
26-07	STC Pump	ES-7193	Rev 1	001	50105-01
27-02	Filter	ES-6888	Rev 1	001	50061-01
28-02	Pressure Lubricators			004	Various
28-03	Pressure Lubricators			002	Various
28-04	Flex Conveyor	ES-7323	Rev 2	001	50126-01
28-06	Ram Valve			001	50164-01
29-02	Separator	ES-7269	Rev 2	001	50116-01
29-04	Gas Superheater	ES-6850	Rev 1	001	50076-01
31-04	Storage Tank	ES-6856	Rev 3	001	50040-01
31-06	Storage Tank	ES-6856	Rev 3	001	50040-01
31-08	Storage Tank	ES-6856	Rev 3	001	50040-01
31-10	Storage Tank	ES-6856	Rev 3	001	50040-01
32-02	Stripper Column	ES-7249		001	50112-01
32-04	TCS Column	ES-7250		001	50113-01
32-06	DCS Column	ES-7251		001	50114-01
32-08	Silane Column	ES-7288		001	50122-01
34-02	Stripper Cond	ES-6802	Rev 2	001	50016-01
34-06	Col Reboiler	ES-6798	Rev 3	001	50015-02
34-08	Column Condenser	ES-6799	Rev 3	001	50019-01
34-10	Col Reboiler	ES-6798	Rev 3	001	50015-03
34-12	Gas Cooler	ES-6828	Rev 2	001	50022-01
34-14	Column Condenser	ES-6799	Rev 3	001	50019-02
34-16	Gas Cooler	ES-6828	Rev 2	001	50022-02
34-18	Column Condenser	ES-6799	Rev 3	001	50019-03
34-24	Gas Cooler	ES-6828	Rev 2	001	50022-03
34-26	Transfer Coil			001	50028-01
35-02	Reactor	ES-6856	Rev 3	001	50040-02
35-04	Reactor	ES-6856	Rev 3	001	50040-03
36-02	LMV 801 Pump	ES-6719	Rev 1	001	50005-02
36-04	LMV 801 Pump	ES-7286	Rev 2	001	50005-03
36-05	LMV 801 Pump	ES-7285	Rev 2	001	50005-04
37-02	Filter	ES-6885	Rev 1	001	50062-01

TAG NUMBER	DESCRIPTION	BM/SPEC NUMBER		QUANTITY ORDERED	P. O. NUMI
441-04	Flaker Feed Bin			001	50033-02
441-06	H_2 Receiver	ES-7278		001	50017-03
443-02	Pyrolysis H2 Compres			001	50011-01
444-02	Gas Cooler	ES-6828	Rev 2	001	50022-04
451-04	Surge Tank	ES-6852	Rev 3	001	50035-08
451-06	Catch Tank	ES-6852	Rev 3	001	50035-09
451-08	Dust Bin	ES-6857	Rev 2	001	50033-03
451-18	4500 Gal Tank	ES-7499	Rev 1	001	50160-01
452-02	Tailing Column	ES-6894	Rev 2	001	50090-01
453-02	Centrif Blower	ES-6714	Rev 2	001	50002-01
453-04	Gas Ind Blower	ES-6715	Rev 2	001	50001-01
453-05	Gas Ind Blower	ES-6715	Rev 2	001	50001-01
454-04	Agglomerator			001	50085
456-08	Gould Pump	ES-6896	Rev 2	001	50091-01
457-04	Dust Bag	ES-7594		001	50187-01
457-06	Filter	ES-6886	Rev 1	001	50060-01
458-04	Drum Packer	ES-6861	Rev 1	001	50073-01
459-02	Scrubber	ES-6862	Rev 2	001	50075-01
459-04	Scrubber	ES-6862	Rev 2	001	50075-02
459-08	Fuel Burner	ES-6883	Rev 3	001	50083-01
459-10	Fuel Burner	ES-6883	Rev 3	001	50083-01
459-12	Fuel Burner	ES-6883	Rev 3	001	50083-01
459-14	Fuel Burner	ES-6883	Rev 3	001	50083-01
459-16	Agitator	ES-7268	Rev 1	001	50115-01
459-18	Immersion Heater	ES-7498	Rev 1	001	50161-01
459-18	Agglometer Cleaner			019	33659-01
459-20	Agitator	ES-7268	Rev 1	001	50115-02
461-02	Expansion Tank	ES-7478	Rev 1	001	50055-01
461-06	350 Gal Day Tank	ES-7477	Rev 1	001	50162-01
464-06	Circ Heater	ES-7346	Rev 1	001	50129-01
466-02	Gould Pump	ES-6893	Rev 1	001	50012-01
466-04	Centrif Pump	ES-7136	Rev 1	001	50013-01
466-05	Centrif Pump	ES-7136	Rev 1	001	50013-01
466-06	Oil Pump	ES-7284	Rev 1	001	50119-01
466-07	Oil Pump	ES-7284	Rev 1	001	50119-01
469-02	Cooling Tower	ES-7205	Rev 1	001	50086-01
469-06	H_2O Treat System	ES-7197	Rev 1	001	50087-01
469-12	Chilling System	ES-6782	Rev 3	001	50088-01
469-14	Instr. Air Pack	ES-7044	Rev 1	001	50092-01
469-16	Heater	ES-6895	Rev 1	001	50089-01
469-20	Instr Air Dryer	ES-7299	Rev 1	001	50125-01
641-02	1500KV Transform	ES-7179		001	50094-01
642-02	480V MCC	ES-7218	Rev 1	002	50093-01
643-02	Emerg Generator	ES-7052	Rev 1	001	50102-01
722-02	Main Instr. Panel			001	50134
725-02	I/O Panel NO1 D	A-2147839	C	001	50220-01
726-02	Scan Panel	A-2146764	B	001	50209-01
726-04	"QA" Panel			001	50223
726-06	"N" Panel			001	50336
728-02	I/O Panel NO2 E	A-2147840	C	001	50219-018
728-04	I/O Panel NO3 F	A-2147841	C	001	50222-019
728-06	I/O Panel NO4 G	A-2147842	A	001	50221-019
Spare	Pressure Lubricators			001	Various

APPENDIX B

PROCESS FUNCTIONAL PACKAGE FOR A

1000 MT/YR COMMERCIAL POLYCRYSTALLINE

SOLAR SILICON PLANT

CONTENTS

TABLES

ILLUSTRATIONS

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1.0 INTRODUCTION

The UCC silane-to-silicon process has proceeded through several stages of development over the last several years in response to the requirements of the funding agency, the Department of Energy (DOE), and the timetable and goals set by the contract management agency, the Jet Propulsion Labs (JPL). As a part of the proposal to build an Experimental Process Systems Development Unit (EPSDU) to demonstrate this process, the contract calls for a periodic economic assessment of the technology on a commercial scale basis to assist JPL's evaluation of this process. The first of these economic assessments, the baseline assessment, is timed to be co-incident with the engineering effort of EPSDU. This timing was chosen to allow the baseline assessment to occur at the earliest possible time while still taking advantage of the knowledge gained in the engineering effort. A second assessment is scheduled to update the baseline assessment at a point where construction of the EPSDU has neared completion and the last free-space pyrolysis and silicon powder consolidation R&D is completed. A final assessment is scheduled after the EPSDU has been operated and actual production data has been collected.

This baseline assessment contains three elements: the process functional package, reported here; an estimate of the cost of such a commercial solar silicon plant, based on the process package; and lastly a determination of the product cost from such a plant based on the utility requirements, manpower requirements, raw material requirements, capital charges, and other production costs. The assessment is to be reported on the basis of a hypothetical plant producing 1000 MT/YR of electronic-grade polycrystalline silicon material for the solar array market. The product cost should include typical charges and costs for a plant constructed within the United States by private investment with a sufficient allowance for profitability to justify such investment.

The baseline functional package includes information to prepare suffi-
cient preliminary engineering to support an estimate: a block flow diagram,
a heat and mass balane, a detailed process description with process flow
diagram, a preliminary overall plant layout, a process equipment list, and
process equipment functional specifications. Other process information, of
a more general sense, is understood to be identical to EPSDU with appropriate
allowances for a permanent facility (as opposed to the temporary nature of
EPSDU) and a more optimal process arrangement. Examples of this other infor-
mation are cleaning requirements, process sealing requirements, painting and
insulation provisions, safety restrictions (such as electrical classifications)
raw materials, and product handling.

1.1 Design Basis

The design basis of this process package is the included process arrange-
ment, to be operated on a production basis of 85% on-stream factor, as measured
on a yearly basis. Raw materials are assumed to be a commercial grade of
98% pure metallurgical-grade silicon, a technical grade of 98% pure silicon
tetrachloride, commercial grade liquified hydrogen gas, and a commercially
available grade of cement copper containing approximately 85% contained copper.
Utilities are assumed to be electrical power, natural gas, potable water,
appropriate cooling water treatment chemicals, commercial liquified nitrogen
gas, and commercial liquified argon gas. The consummables are assumed to
be replacement quartzware (for pyrolysis/consolidation), replacement redistri-
bution catalyst (Rohm & Haas A-21) and appropriate quality control supplies.
Other miscellaneous chemicals are assumed to be start-up solvents (methanol
and hexane), laboratory chemicals, lime for waste neutralization, replacement
refrigerant, and replacement heat transfer oil. The plant product and by-
products are assumed as polycrystalline silicon shot of electronic quality,
low-grade (asphalt or glue bulking quality) fumed silica and 20% (pickling
strength) muriatic acid. The plant wastes are gases disposed of through
atmospheric vents of suitable quality to meet federal guidelines, process and
sanitary wastewater of suitable quality to discharge to a municipal water
treatment plant, solid waste and trash capable of being disposed of at a non-
hazardous material installation, and thermal emissions in the form of a cooling
tower plume.

1.2 Assumptions

1.2.1 General

a. Plant is designed and built by UCC to Linde Division Standards by Gas Products Engineering at a hypothetical site in the Chicago metropolitan area.

b. The plant site is a clear and unobstructed site abutting an existing UCC facility. Utilities such as potable water, electricity, and sewer lines are available at the plant battery limits.

c. The process design activity is begun in the 2nd quarter of 1983 and the plant start-up is commenced in the 1st quarter 1986.

1.2.2 Process

a. The major process equipment will remain functionally identical to EPSDU, with a scaling factor, with the exception of waste treatment and pyrolysis/consolidation.

b. The waste treatment area will be functionally identical to the original EPSDU process, presented in EM-6429, June 1, 1979, "Process Design Package for a 100 MT/YR Silane-to-Silicon Plant", with a scaling factor. Silica will be packed into 100 cubic feet, aluminum, transportable "tote" bins (trademark of Tote Division of Hoover Ball and Bearing Corp.) instead of 55-gallon drums.

c. The pyrolysis/consolidation area is conceptualized based on in-hand information. It assumes a pyrolysis reactor based on a 30-inch quartz liner which is scaled up from the Tonawanda 8-inch quartz liner. The 8-inch reactor is given a capacity rating of 5 lbs/hr of silane. Scale-up is done based on cross-sectional area and aspect ratio to yield a capacity of 80 lbs/hr of silane for 30-inch quartz lined unit. It is assumed that the silicon powder can be dense-phase conveyed, however, occasional flakes may result from semi-solids formations in the reactors. This

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flaking off is assumed to be small enough in frequency not to
effect the mass balance or equipment sizing. The melter is
assumed to have a capacity of 15 kg/hr of silicon and produce a
tight distribution of nominal 2 mm diameter shot. The shot is
assumed to "skin over" sufficiently in 10 feet of free-fall
through hydrogen to allow final cooling in a mildly fluidized
bed. The amount of cooling in the fluidized bed is assumed to
be roughly half of the requirement. The cooled shot is assumed
to require inert gas padding for shipment, but is capable of
being shipped in plastic lined 55-gallon drums.

1.2.3 Raw Materials, Utilities, Products

Metallurgical-grade silicon is assumed to be delivered in bulk using a
cleaned concrete truck with 40,000 lbs gross weight, with an on-board blower
for discharge into a 4" standard fitting (per phone conversation with Matlack
trucking office). The metallurgical silicon arrives ground to a nominal 300
micron size and is assayed to be 98% contained silicon with the remainder being
predominantly iron and aluminum impurity (similar to the grade received at UCC's
Sistersville facility). The technical-grade silicon tetrachloride is assumed to
be delivered in a 4000-gallon tank truck, fitted for nitrogen-pressure unloading.
The STC is clear and assayed to at least 98% silicon tetrachloride with the
remainder being predominantly trichlorosilane and dimethyl-dichlorosilane
(similar to that produced at Sistersville). Hydrogen, nitrogen and argon are
delivered as liquified gases by appropriate sized tank trucks and are of a
commercial quality equal to that produced by UCC, Linde Division. Cement
copper is received in one-to-five gallon pails in lots suitable for truck
transportation and is of a quality similar to that used at Sistersville, con-
taining 85% copper.

Silicon shot product is assumed to be shipped by truck in 10 to 20 ton
lots of 55-gallon drums. Fumed silica is assumed to be shipped in returnable
110 cubic feet capacity aluminum bins owned by UCC. Muriatic acid is assumed
to be shipped by tank truck in nominal 2000-gallon lots by a shipper such as
Chemical Leman. Neutralized waste solids are assumed to be removed and trans-
ported to a low quality refuse disposal in unlined 55-gallon closed drums in
lot sizes suitable for truck transportation.

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Electrical power, potable water and sewer connection are assumed to be of a quality or nature similar to that of EPSDU.

1.2.4 Yard and Auxiliaries

In addition to the main process equipment, the plant is assumed to contain the following yard and auxiliary improvements: a switch gear building with emergency power generation, a two-cell cooling tower, a parking lot for 50 cars, an office building with first aid station, a maintenance building, a building for personnel activities containing a shower room, eating area and locker room, a laboratory/quality control building, a fire-fighting capability based on Kiddle High-expansion foam similar to that used at EPSDU, a shipping building attached to the pyrolysis/melter building, and an external weather-protected storage pad for solvent, acids, and drum chemical storage. The control room should be outfitted for supervisory/distributed computer control using localized pneumatic/electronic instrumentation similar to a Taylor MOD III or Honeywell TDC-2000. A dedicated data collection computer with extensive Scan-i-valve input, such as used for EPSDU, is not required. The HVAC requirements should be of a quality to maintain a 65^OF winter temperature, and a reasonably ventilated summer condition for maintenance and shipping areas, and a 65^OF winter temperature and 80^OF summer temperature for personnel activity areas. The number of air changes in the pyrolysis/melter building should be suitable for unclassed electrical equipment. The metallurgical silicon and fumed silica handling should be in a building maintained at 50^OF in the winter with summer ventilation suitable for a dusty area.

2.0 BLOCK FLOW DIAGRAM

A block flow diagram is included as Figure 1 for familiarization with the process. For more detailed process schematics, see the process flow diagram and process description in the following section.

The process proposed for preparing semiconductor-grade silicon from metallurgical-grade (M-G) silicon is based on a well-integrated arrangement of purification steps that provides a cost-effective process system. The three basic steps entail converting M-G silicon to trichlorosilane, redistributing the trichlorosilane to produce silane, and thermally decomposing the silane to form polycrystalline silicon powder. The powder is then melted and consolidated in a shotting apparatus to yield polycrystalline silicon shot of nominal 2 mm size.

Raw materials for the process consist of M-G silicon (98%-pure) and copper catalyst; hydrogen and silicon tetrachloride must be added to replace the hydrogen and chlorine lost from recycle loops in the form of process wastes. The wastes to be disposed of include gaseous light impurities and heavy impurities composed of metal chlorides. Except for the raw material feed, waste disposal, and silcion-product streams, the entire process — hydrogenation, distillation, redistribution and separation — is a closed-loop system in which purification is accomplished in each major step as process fluids are being recycled through the equipment. The process is shown in block-diagram form on Figure 1. The major steps are summarized in the paragraphs that follow.

M-G silicon and cement-copper catalyst are batch-fed to the hydrogenation reactor which also receives the hydrogen and silicon tetrachloride recycle streams. The silicon is consumed to make trichlorosilane in an equilibrium reaction:

$$Si + 2H_2 + 3SiCl_4 \overset{Cu}{\underset{\rightleftarrows}{}} 4HSiCl_3$$
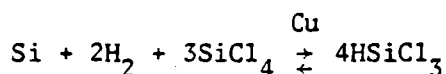
Metallic impurities in the silicon are converted to chlorides. Hot off-gases are quenched to their dewpoint to reject metal chloride impurities, and then are condensed to ambient temperature. The non-condensible gases, predominantly hydrogen, are reheated and recycled to the reactor.

The condensed crude trichlorosilane, containing about 80% silicon tetrachloride and some dissolved gases, is fed to the first of four distillation columns. In this column, gaseous light impurities are removed to part-per-billion level. In the second column, the bulk of the silicon tetrachloride is separated from other chlorosilanes and then is heated and recycled to the hydrogenation reactor. The other chlorosilanes from the second column are fed to the third column where trichlorosilane is separated from dichlorosilane.

The trichlorosilane bottoms product from the third column is redistributed to dichlorosilane and silicon tetrachloride in an equilibrium reaction using an amine-based resin catalyst:

$$2HSiCl_3 \rightleftarrows H_2SiCl_2 + SiCl_4$$

The redistribution products are recycled to the second column for separation.

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The dichlorosilane distillate from the third column is redistributed to silane, monochlorosilane, trichlorosilane, and silicon tetrachloride in three reactions:

$$2H_3SiCl \rightleftharpoons H_2SiCl_2 + SiH_4$$

$$2H_2SiCl_2 \rightleftharpoons HSiCl_3 + H_3SiCl$$

$$2HSiCl_3 \rightleftharpoons H_2SiCl_2 + SiCl_4$$

The redistribution products are then fed to the fourth column for separation.

In the fourth column, silane is separated from the other process compounds which are returned to the third column. Since gases lighter than dichlorosilane were removed in the first column, and remaining traces of diborane, which is closest in boiling point to silane, are removed to less than part-per-billion levels in the fourth column, the silane distillate is extremely pure. It is collected, tested for purity, and vaporized by heating to ambient temperature before being pyrolyzed.

In the pyrolysis reactor, the silane is expanded in a nozzle and is irreversibly decomposed into silicon powder and hydrogen according to:
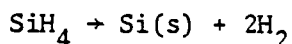
$$SiH_4 \rightarrow Si(s) + 2H_2$$

The hydrogen by-product is removed from the fine silicon powder and recycled to the hydrogenation section. The silicon powder is batch-fed into a heated quartz crucible where it is melted. The molten silicon is then dropped in a shotting apparatus to yield a nominal 2 mm spheroid and cooled to near ambient temperature before being removed as the final product of the process.

The gaseous and liquid process wastes are gathered in headers and processed for disposal. Wastes are thermally oxidized in a burner and then are scrubbed free of silicon and hydrogen chloride. The waste by-products are scrubbed exhaust gas, silica powder, and muriatic acid.

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Light Impurities
To Disposal

Recycle
Hydrogen

| Quench and Condense | STC TCS* Light impurities | First Distillation Column | | Redis-tribution DCS | TCS DCS Silane* MCS STC | Fourth Distillation Column |

Silane liquid

Heavy Impurities (Chlorides) to Disposal

STC*
TCS*

DCS*
MCS
TCS

TCS*
DCS
MCS
STC*

| Second Distillation Column | TCS* DCS* STC | Third Distillation Column |

Silane Vaporization

H₂
STC
TCS
Impurities

H_2
STC
TCS
Impurities

Silane Vapor

TCS*
STC*

M-G Silicon

Copper Catalyst

| Hydro-genation Reactor | STC Recycle | | | TCS STC DCS | Redis-tribution TCS |

Silane Pyrolysis

Make-up STC

Recycle Hydrogen

| Powder Separation | Silicon Powder H_2 |

Make-up Hydrogen

Silicon Powder

| Melter and Shotting Tower | Polycrystalline Silicon Shot |

LEGEND	
DCS	Dichlorosilane
MCS	Monochlorosilane
STC	Silicon Tetrachloride
TCS	Trichlorosilane
*	designates primary process fluid

FIGURE 1

SILANE-SILICON PROCESS - BLOCK FLOW DIAGRAM

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3.0 PROCESS DESCRIPTION/PROCESS FLOW DIAGRAM

This process description should be read in conjunction with the process flow diagram, F-2073005, Alt. B., (Sheets 1-5). The stream numbers used in the description are used in the stream catalog (Section 4). The equipment numbers used in the description are those used on the following equipment list (Section 6) and the functional specifications (Section 7).

The process design converts 98%-pure, commercial, metallurgical-grade silicon into ultra high-purity silicon metal suitable for solar-cell applications. The feed metallurgical silicon is converted to gaseous trichlorosilane (TCS) in a hot, copper-catalyzed, fluid-bed reactor. Non-volatile, metal-chloride contaminants are separated from the reactor product stream by removal of a small, liquid fraction at its dewpoint, and hydrogen is subseqeuntly separated as a noncondensable gas from the condensed crude TCS liquid. The crude TCS liquid is stripped free of dissolved volatile impurities in a reboiled stripping column. The material is then processed through a series of three conventional distillation columns and two redistribution reactors. This equipment progressively replaces chlorine in the chlorosilane molecules with hydrogen, pushing the heavily chlorinated compounds back to the front of the process and hydrogen-rich molecules forward. Ultimately, silane is produced and subjected to a final distillation in the fourth column that yields an ultra-high purity, liquid-silane product.

The pure silane is pyrolyzed to form hydrogen gas and high-purity silicon metal powder. The powder is consolidated by melting and removed from the process.

Reference to the plant process flow diagram is suggested as necessary for following the ensuing detailed process description.

3.1 Hydrogenation

Ninety-eight percent pure powdered, metallurgical-grade silicon, having an average particle size of 300 microns, is received by tank truck and is transferred pneumatically (100) to the metallurgical-grade silicon storage bin, 411-02 by the truck's on-board blower. The conveying air is passed through the metallurgical-grade silicon unloading filter, 417-02, before being

exhausted to the atmosphere. Silicon powder from the storage bin is batch
fed to the hydrogenation catalyst blender, 428-02, into which a small
amount of copper catalyst (101) is also being added. The catalyst/silicon
powder blend is transferred by a conveyor, 428-04, to the metallurgical-grade
silicon lock hopper, 421-02. Hydrogen gas (105) is used to pressurize the
lock hopper to 550 psia after it has been purged free of air with utility
nitrogen (1400). Lock hopper outlet valving is then opened and the silicon/
catalyst mixture is gravity-fed into the fluid-bed, hydrogenation reactor, 425-02
operating at 932°F and 515 psia. A recycle stream of hydrogen (122) and sili-
con tetrachloride (STC) (123), heated to 961°F, is controlled such that the
streams entering the reactor contains equal molar proportions of hydrogen and
STC. Make-up hydrogen (104) is added to the hydrogenation reactor from
pyrolysis recycle or hydrogen storage.

In the hot (500°C), fluid-bed environment of the reactor, the STC is
hydrogenated according to the overall reaction,

$$3SiCl_4 + 2H_2 + Si \underset{\leftarrow}{\overset{Cu}{\rightarrow}} 4HSiCl_3,$$
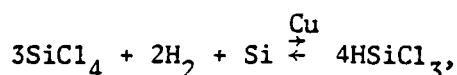
which is reversible and which, in the presence of copper catalyst and
selected bed dimensions, proceeds nearly to equilibrium to yield a hot, over-
head, vapor stream (108) that contains about 15% TCS, 44% unreacted hydrogen
and 41% STC. This hot vapor also contains some unreacted silicon dust, cop-
per powder, and all the trace metal contaminants released from the metallurgi-
cal silicon in the reactor as metal chlorides. This stream is quenched to its
dewpoint, about 180°C, by a large STC/TCS liquid recycle stream (111) in an
efficient venturi and solids removal contactor 429-02. The two-phase product
(110) is delivered to the waste settler tank, 421-04. Liquid STC/TCS from the
settler tank is pumped by the quench contactor pump, 426-02, to the venturi
quench contactor. Many of the metal contaminants form insoluble complexes with
one another in this unit (e.g., aluminum chloride forms a complex with iron
chloride) and settle to the bottom of the quiescent settler tank as sludge.
A small stream (112) of STC/TCS liquid from the settler containing unreacted
silicon powder dust, copper catalyst dust, complexed metal salt sludge and dis-
solved metal salts is rejected through the waste chlorides tank, 421-06, to
waste disposal.

The dust-free STC/TCS hydrogen stream, with depleted metal chloride contaminants, flows from the settler through a double shell quench condenser, 424-02, which condenses the STC/TCS from the noncondensable unreacted hydrogen to the quench condenser receiver, 421-08. The hydrogen gas (118) is separated from the STC/TCS condensate, increased in pressure by the recycle hydrogen compressor, 423-02, to 522 psia, and heated to $520^{\circ}C$ in the recycle gas superheater, 429-04, before being recycled to the fluid bed STC hydrogenation reactor (122). A small quantity of liquid STC/TCS (115) is circulated from the quench condenser receiver to the quench condenser by the quench condenser wash pump, 426-04. This liquid is sprayed on the tube walls of the quench condenser to wash away any metal chloride contaminants that may have carried over from the settler tank and been deposited. Liquid STC/TCS is also supplied by gravity from the quench condenser receiver to the settler tank (116) as required to maintain a constant liquid level. The latent heat in this stream provides the cooling needed to reduce the temperature of the reactor product from the reactor outlet temperature of $500^{\circ}C$ to the settler operating temperature of approximately $180^{\circ}C$. Crude STC/TCS product (119) at ambient temperature is delivered to the large, crude TCS storage tank, 421-10, operating at 100 psia. This nitrogen-pressurized tank provides decoupling between the STC hydrogenation and the distillation/redistribution/purification train. Shutdown of either plant section will not necessitate immediate outage of the other because of this feature.

3.2 Distillation/Redistribution

Crude STC/TCS liquid flows (125) from the crude TCS storage tank, 421-10, to the stripping column, 432-02, which operates at 70 psia and is designed to remove the 99% of the possible hydrogen sulfide dissolved in the feed. The stripping column is designed to accomplish two objectives: to remove all volatile contaminant gases lighter than silane to at least 0.01 PPB and to remove 99% of the heaviest of the volatile contaminants. These contaminants are heavier than silane. It is not necessary to drive the stripper when removing the volatile contaminants heavier than silane since they will be rejected in the bottoms of the silane column, 432-08, and purged (226). The reboiler on the stripper, operating at $120^{\circ}C$, is driven by hot oil from a closed-loop, hot-oil heating system; heat is extracted from the overhead condenser by refrigeration. Overhead distillate vapor containing the rejected light gases is directed to waste incineration. Since the feed to the column contains significant

amounts of DCS, column operating conditions have been selected to minimize overhead losses of this valuable material.

Crude STC/TCS liquid, free of dissolved volatile contaminants, flows (206) from the stripper reboiler, 432-02, to the TCS column, 432-04. The TCS column receives a second feed stream (212) containing a mixture of chlorosilanes including STC that has been recycled back from the reboiler, 434-10, of the DCS column. Operating at 55 psia, the TCS column is designed to recover 97% of the total STC in its two feeds in the bottom liquid product, and 97% of the total TCS feed in its overhead distillate product. The STC bottom liquid product (238) is cooled by the STC cooler, 434-24, against cooling water and delivered to a low-pressure, liquid STC storage tank, 421-12. Liquid STC from this tank (127) is pumped through the hot end of the quench condenser, 424-02, which preheats the STC, through the recycle STC vaporizer, 424-04; heated to $520^{\circ}C$ in the recycle gas superheater, 429-04; and recycled back to the fluid-bed reactor (123). To make up for the small amounts of chlorine that are rejected from the settler tank (112) and the stripper column overheads (2501), technical-grade STC is periodically added (102) to the inventory in the STC storage tank.

The trace-metal contaminants (PCl_3, $AsCl_3$, $FeCl_3$, $NiCl_2$, $CuCl_2$, $CaCl_2$, etc.) that enter with the crude STC/TCS liquid fed to the TCS Column (206) have a higher boiling point than STC and will, therefore, be rejected with the bottoms product (238) back to the hydrogenation reactor through the STC vaporizer and superheater, and end up as sludge in the settler tank. If present, contaminants lighter than STC (B_2H_6, PH_3, AsH_3, BCl_3, etc.) will leave the STC column with the distillate product.

The TCS column overhead vapor (209), a mixture of MCS, DCS, and TCS, is totally condensed against cooling water in the TCS column condenser, 434-08. This liquid is pumped and divided between column reflux (213) and distillate product (214). The distillate product flows to the DCS column, 432-06. The DCS column, operating at 170 psia, receives a second feed (225) recycled from the reboiler, 432-08, of the silane column. Both feeds contain mixtures of chlorosilanes. Column operating conditions have been selected to deliver 97% of the total TCS contained in both feeds to the bottom and 97% of the DCS feed

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to the top of the column as distillate product. The bottom product leaves (235) the hot-oil driven DCS column reboiler, 434-10, and is cooled to 70°C. It is fed (215) liquid-phase through an Amberlyst® A-21, amine base, ion-exchange resin, catalytic-redistribution reactor, 435-02, before being recycled (212) back to the TCS column. The reactor catalyzes the following reactions:

$$2HSiCl_3 \overset{A-21}{\rightleftharpoons} H_2SiCl_2 + SiCl_4$$

$$2H_2SiCl_2 \overset{A-21}{\rightleftharpoons} H_3SiCl + HSiCl_3$$

$$2H_3SiCl \overset{A-21}{\rightleftharpoons} SiH_4 + H_2SiCl_2$$
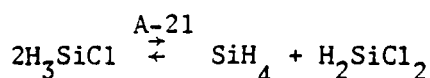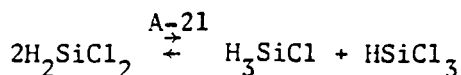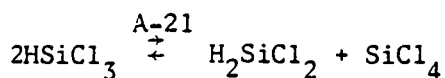
The reactor is sized to convert its feed, which is predominantly TCS, to 95% of its equilibrium value. The equilibrium of these reactions is such that only small quantities of silane and MCS are formed while substantial quantities of DCS and STC are produced.

Overhead vapor (219) from the DCS column is totally condensed against cooling water in the DCS column condenser, 434-14, before being pumped and divided between reflux (220) and distillate product (222). This distillate product is cooled to 50°C against cooling water by the DCS cooler, 434-16, before being passed, liquid-phase, through a second redistribution reactor, 435-04, similar to the unit previously described. The feed to this reactor, primarily DCS, is redistributed to form a spectrum of chlorosilane and silane that is consistent with the equilibrium reactions listed above. Silane, the ultimate product of this distillation/redistribution train, increased from 0.44 to 11.45 mole percent in passing through the reactor. This redistribution reactor also acts as a trap for residual boron impurities (BCl_3, B_2H_6), which combine irreversibly with the amine-resin catalyst.

The reactor product (224) is fed directly to the silane column, 432-08, operating at 360 psia. Operating conditions have been selected to permit rejection as bottoms product all material except silane. The reboiler, 432-08, is driven by hot oil and heat is removed by the silane column condenser, 434-18, by refrigeration. Volatile contaminants heavier than silane are not

completely removed in the stripping column and therefore appear in the bottoms product. The most volatile of the potential contaminants (i.e., the contaminant having the lowest boiling point) is diborane, B_2H_6. Its normal boiling point is $-93^{\circ}C$, while silane boils at $-112^{\circ}C$ and MCS at $-31^{\circ}C$. Operating conditions for the column have been chosen to separate silane from diborane and all other higher-boiling contaminants. The column has a calculated ability to produce a silane distillate product containing <0.01 PPB diborane when the column feed contains 5 PPB. Ninety-seven percent of the silane contained in the column feed is removed as an ultra-high-purity overhead liquid product (231). This liquid silane is directed to one of four shift tanks. When the selected shift tank is filled, it is isolated and checked for purity and another tank is placed into service. Refrigeration is supplied to these tanks, as required, to compensate for ambient-heat leak and to prevent pressure build-up and loss of contents. When quality checks verify adequate purity, the liquid in the tank is vaporized at a controlled rate by raising the refriger-ant temperature to maintain a constant tank pressure while the vapor is re-moved from the top of the tank (233). The vapor is then sent (235) to silane pyrolysis equipment for the production of the final, high-purity, silicon metal product. At any one time, one shift tank is receiving product liquid silane from the silane column, quality control analyses are being made on a second unit, and the third tank is supplying silane to pyrolysis equipment. A fourth tank is available in the event the silane does not pass the quality control analyses; it can be used to accept product liquid silane while the silane that did not pass the quality check is being drained.

All materials entering the silane column, other than silane, are removed as bottoms liquid product (239) consisting primarily of DCS and TCS with small amounts of MCS and STC. This liquid stream is returned to the DCS column.

The possibility that certain contaminants of intermediate volatility could become trapped in sections of the distillation system has been recognized. For example, BCl_3, whose boiling point falls between those of DCS and TCS, will re-port with the overhead distillate product (214) in the TCS column, enter the DCS column, and be rejected to its bottoms product (236). The bottoms product, however, recycles back to the TCS column. Therefore, conceivably, the concentra-tion of BCl_3 could build in the system to the level necessary either to force it

out the top of the DCS column or the bottom of the TCS column (assuming the material is not irreversibly trapped by the TCS redistribution reactor, 435-02). To prevent impurity buildup, provision has been made to recycle a small bleed stream (237) from the bottom of the DCS column back to the STC tank. Similarly, contaminants whose boiling point fall between those of DCS and silane (e.g., B_2H_6, PH_3) could build in the liquid streams (222, 225) traveling between the DCS column and the silane column. To avoid this possibility, provisions have been made to bleed a small quantity of liquid from this system back to the STC tank. There is also the possibility that contaminants heavier than STC are present in the STC storage tank. These contaminants could build up in the STC recycle vaporizer, 424-04, which would be blown down to the waste settler by a temporary connection, if required. In all cases, the bleed streams are designed to provide an exit path from the system.

Boron is the most undesirable contaminant in the silicon metal used for electronic and semiconductor applications because it is electrically active and difficult to remove by solid-silicon, zone-refining techniques because of its unfavorable liquid/solid distribution coefficient. Three methods of boron removal have been incorporated into the process design of this plant:

- Small amounts of silane (240), which may react with any BCl_3 that is present and form B_2H_6, are fed to the bottom of the stripper. The B_2H_6 will leave with the stripper overhead product as opposed to BCl_3 which leaves with the stripper crude bottoms product.

- of product chlorosilanes through catalytic redistribution reactors will result in the trapping of residual boron by this catalyst.

- Finally, the silane distillation column is designed and operated to reject from the silane product any or all diborane that may be contained in its feed.

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Any of these processing steps are capable of removing boron from the system to acceptable levels. There is conservatism and redundancy built into the process to assure complete boron removal.

3.3 Silane Pyrolysis/Consoldiation

Ultra-high-purity silane vapor flows (233) from the top of one of the silane storage tanks, 431-04, and is throttled to about 30 psia. The silane is passed through a micropore filter, 437-02, and injected by water-cooled nozzle (300) into six quartz-lined, free-space, silane-pyrolysis reactors, 445-02. Each reactor is heated by a resistive heater element on the outside of the reactor, and the wall temperature is maintained at $960^{\circ}C$. The gas in the reactor is convectively heated to about $760^{\circ}C$; it decomposes exothermically to form silicon and hydrogen gas according to the reaction;

$$SiH_4 \rightarrow Si + 2H_2$$
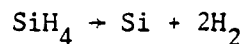
The hydrogen produced is separated from the silicon powder by porous metal filters located in the lower chamber of each reactor. The hydrogen collected (311) then flows to a heat exchanger, 444-02, where it is cooled to $100^{\circ}F$ against cooling water. The cooled hydrogen is then compressed to the hydrogenation reactor pressure by a two-stage diaphragm compressor, 443-02, for subsequent recycle to hydrogenation. Mismatch in operating rate between pyrolysis and hydrogenation is balanced by either drawing hydrogen from a liquid hydrogen storage tank, 411-06, or by venting off excess hydrogen to the waste burners. These adjustments to the hydrogen flow are made after the first compression stage to maintain the first stage hydrogen quality at the electronic level. This hydrogen is required for use in purging and fluidization at the pyrolysis reactor, and can therefore be used without fear of contamination from outside sources.

The silicon powder exiting the lower chamber of the pyrolysis reactors (303) is then dense-phased conveyed to a central silicon powder hopper, 441-12. This water-cooled hopper has an internal screen with 1/16" opening to remove any oversize silicon solids that are formed in the pyrolysis reactors. This is done to protect downstream powder feeding equipment. The powder is then fed batchwise to any of 12 melter/shotting assemblies. The powder transfer is

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conveyed using argon or hydrogen pressure to overcome piping resistance. The control over this cycled transfer is achieved by a local microprocessor.

Each melter/shotting assembly (449-04) consists of a small powder bin which receives periodic refill from the central powder hopper, 441-12; a twin-flight screw feeder which feeds the silicon powder at a controlled rate from the small powder bin to the melting chamber; a resistance-heated, quartz-crucible melting furnace based on a Hamco CG-2000; which forms a stream of molten shot by forcing silicon through one or more orifices in the bottom of the apparatus; and a water-jacketed shotting tower where the molten silicon shot is allowed to a free-fall through hydrogen gas to "skin over" the shot followed by a soft landing and subsequent cooling to about 70°C in a slightly fluidized bed. The cooled silicon shot is continuously removed from each shotting tower by a rotary valve, 448-08 which admits the shot to a shrouded central conveyor, 448-06. These rotary valves form a gas seal between the hydrogen atmosphere shotting towers and the nitrogen atmosphere conveyor. The side plates of the rotary valves are additionally tapped to allow the pockets to be atmosphere exchanged as the valve meters the shot to the conveyor.

The twelve melter/shotting assemblies, with the respective rotary valves are arranged in a line of six units on either side of the central shot conveyor. Each line of six units is served by a blower, 443-04, to recirculate cooling hydrogen through the lower fluidized portion of the shotting towers; a filter 447-02, to remove silicon fines from the cooling hydrogen circuit; and a water-driven cooler, 444-04, to cool the recirculated hydrogen gas. These recirculating hydrogen loops operate between 65°C and 35°C and are controlled to maintain a slightly fluidized condition inside the lower portion of the shotting towers and a pressure of 15.2 to 15.7 psia.

The silicon shot is conveyed to an enclosed nitrogen-atmosphere silicon shot bin, 441-08, which holds up to one day's production. Nitrogen is added to the bottom of the bin through mesh pad distributors and flows up through the shot to finish the cooling process and maintain an inert atmosphere. The nitrogen flows from the top of the bin, down the conveyor and removed at the other end of the conveyor. This nitrogen flow is then recirculated back to

the bottom of the shot bin by a central blower, 443-08. The shot is periodically removed from the bottom of the bin and loaded into plastic-lined 55-gallon drums for shipping. Drums are weighed by a scale, 448-10, as they are being filled and tagged as to lot number, net weight and date of manufacture. Samples of each lot are taken for quality control testing.

On the average five pyrolysis reactors and eleven melters are required for meeting capacity. The remaining units are spares to allow for rebuilding.

3.4 Waste Disposal

Waste streams are disposed in an environmentally acceptable manner by high temperature flame hydrolysis of chlorosilanes in (commercial burners) to form hydrogen chloride gas and a fluffy silica solid, known commercially as fumed silica. These reaction products are removed from the combustion gases by the combination of a bag filter and various wet scrubbers. The cleaned combustion gases are then vented to the atmosphere by a blower.

Process wastes are divided into four collection headers: potentially combustible gases containing hydrogen, but no silane or chlorosilanes (2299); chlorosilane gases with more chlorine than hydrogen (2499); silane/chlorosilane gases with more hydrogen than chlorine (2599); and chlorosilane liquids (406). The silane/chlorosilane-free gases are passed through a bag filter, 457-04, before venting to atmosphere. Similarly a bag filter, 417-02, is located over the metallurgical-silicon storage bin, 411-02, to collect silicon fines before venting. The fines from these filters can either be processed through the hydrogenation reactor or disposed of as trash.

Chlorosilane-bearing gases (404) are combusted to yield hydrogen chloride and silica in a waste burner, 459-10. Silane- or hydrogen-bearing gases (2500) are combusted in a separate burner, 459-08. Liquid wastes are collected (406) and combusted in an atomizing, waste burner, 459-14. The various burners all discharge into a vacuum collection system together with enough excess air to quench the flames. To convert all chlorinated material to hydrogen chloride, hydrogen funtionality is added, where necessary, in the form of natural gas.

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To minimize atmospheric venting of chlorosilanes as a result of process upsets, overpressure relief lines are manifolded to a pressure-relief catch tank, 451-06, which phase-separates and feeds material to the appropriate burner for disposal.

Combustion gases from the burners are cooled in the silica agglomerator, 459-16, to about 150-200°C. Silica particles formed in the combustion process agglomerate when cooled in this fashion to form larger, more easily controlled particles. The fumed silica, along with metal oxides formed in the flame, are collected by bag filters, 457-06 and 457-08. Trace sub-micronic solids remaining in the combustion gases (421) are removed by a high-energy venturi, 459-02, driven by a medium-strength, muriatic solution. As the combustion gases pass through the venturi, they are cooled to near dewpoint by humidification. The gases, cleaned of solids, are then scrubbed free of hydrogen chloride with a two-stage venturi ejector, 459-04 and 459-06, producing a nominal 20% muriatic acid solution. The scrubbed gases, cleaned of hydrogen chloride to low levels, then pass through the muriatic tailing column, 452-02, where the gases are neutralized with a lime solution. The gas that is exhausted to the atmosphere (443) by the waste induction blower, 453-04, has solid-particulate and hydrogen-chloride levels that are well below regulated limits.

The solids collected by the bag filters, primarily fumed silica, are discharged through a cyclone, 458-02, and an airlock to the silica dust bin, 451-08. The silica dust (432) is periodically packed into transportable bins, 451-17 using a vibrating table, 458-04, to densify the dust.

The high-energy venturi, 459-02, is purged (426) of collected solids whenever the solids concentration increases to about 2%. Approximately 500 gallons of liquid is purged into a neutralizing tank, 451-10, equipped with a sand filter where it is neutralized with lime, causing solid precipitation. Leachable calcium chloride is filtered from the solids by gravity percolation through the sand filter and is released to the sewer.

The fumed silica by-product from the bag filters is marketable as a low-performance filler for asphalt and glue bulking. The medium-strength, muriatic acid is marketable for such applications as pickling metals.

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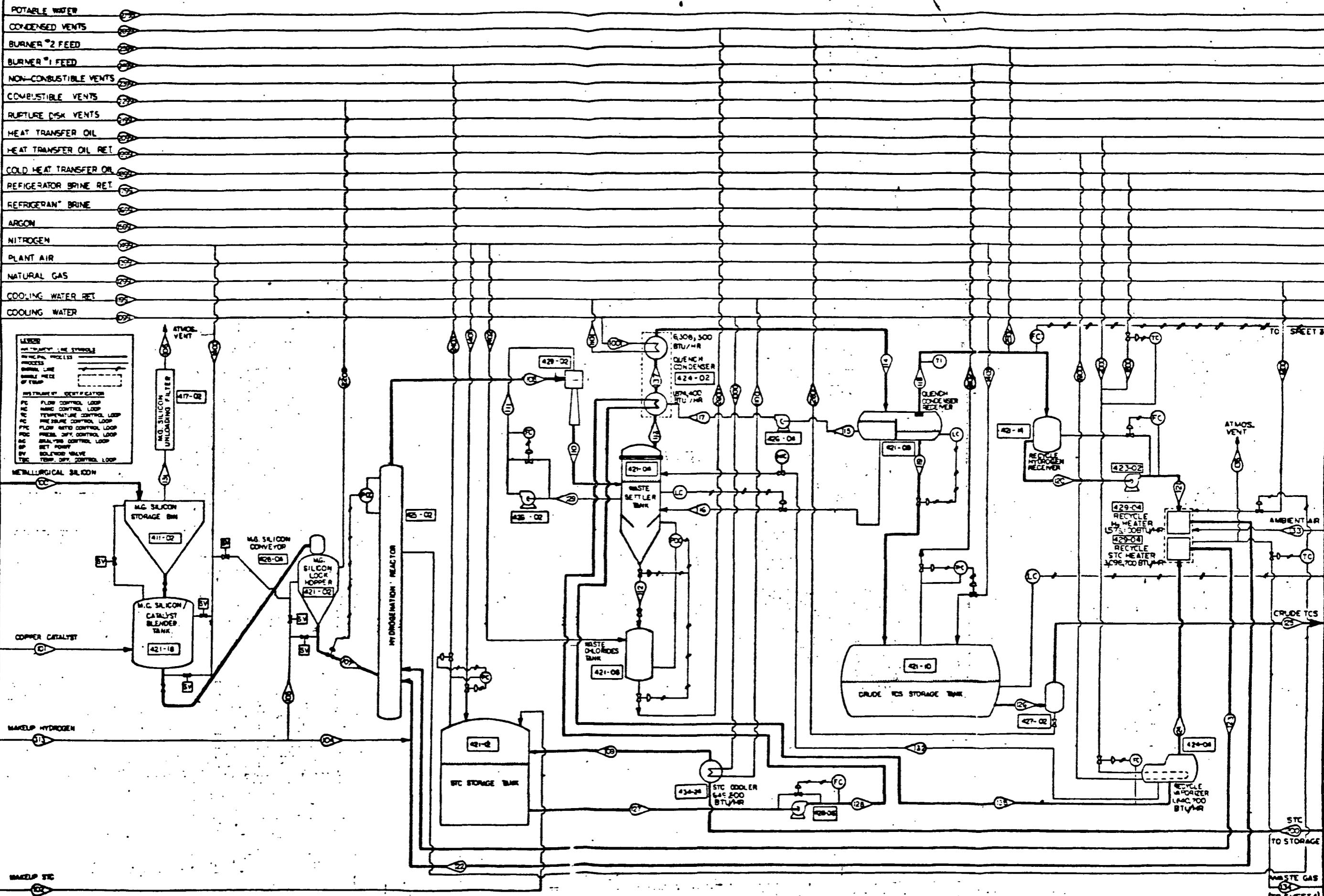
SECTION 1: HYDROGENATION

1000 MT/YR COMMERCIAL PLANT
PROCESS FLOW DIAGRAM

F-2073005

SECTION 2: DISTILLATION/REDISTRIBUTION

UNION CARBIDE CORPORATION

1000 MT/YR COMMERCIAL PLANT
PROCESS FLOW DIAGRAM

F-2073005

SECTION 3: PYROLYSIS CONSOLIDATION

1000 MT/YR COMMERCIAL PLANT
PROCESS FLOW DIAGRAM
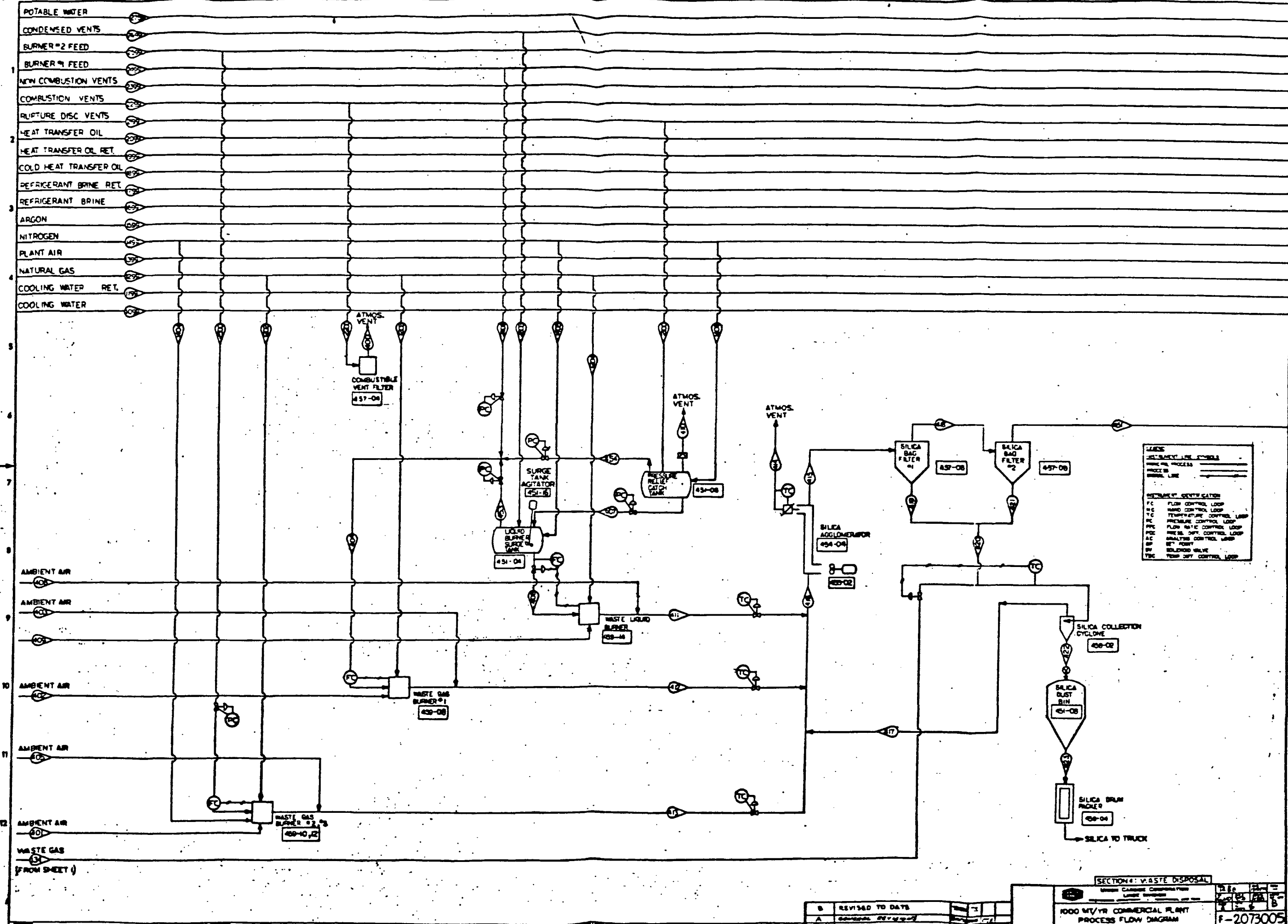
F-2073005

SECTION 4: WASTE DISPOSAL

1000 MT/YR COMMERCIAL PLANT
PROCESS FLOW DIAGRAM

F-2073005

POTABLE WATER
CONDENSED VENTS
BURNER #2 FEED
BURNER #1 FEED
NON-COMBUSTIBLE VENTS
COMBUSTIBLE VENTS
RUPTURE DISK VENTS
HEAT TRANSFER OIL
HEAT TRANSFER OIL RET
COLD HEAT TRANSFER OIL
REFRIGERATOR BRINE RET.
REFRIGERANT BRINE
ARGON
NITROGEN
PLANT AIR
NATURAL GAS
COOLING WATER RET
COOLING WATER

STRONG MURIATIC SCRUBBER
450-04

STRONG MURIATIC PUMP TANK
451-14

SILICA VENTURI
459-02

WEAK MURIATIC SCRUBBER
450-06

WEAK MURIATIC PUMP TANK
451-14

MURIATIC TAILING COOLING
452-02

WASTE SILICA NEUTRALIZER
451-10

MURIATIC ACID STORAGE TANK
451-18

454-08

456-04

456-08

454-02

ATMOS. VENT

453-04

MURIATIC ACID BY-PRODUCT
LIME
SLUDGE TO DISPOSAL
LEACHABLE CHLORIDES TO SEWER

LEGEND

INSTRUMENT LINE SYMBOLS
PRINCIPAL PROCESS
PROCESS
SIGNAL LINE

INSTRUMENT IDENTIFICATION

| B | REVISED TO DATE | | |
| A | | | |

4.0 HEAT AND MASS BALANCE

The heat and mass balance for the commercial process is contained in the following pages. The equipment and stream numbers correspond to the process flow diagram presented in the previous section.

4.1 Heat Duties

The listing of heat duties presented in Table I is a complete listing of the thermal requirements of the heat exchangers in the process.

TABLE I

1000 MT/YR COMMERCIAL PLANT HEAT DUTIES (ISSUE 3)

Equipment No.	Equipment Name	Heat Duty (Btu/Hr)
424-02	Quench Condenser	8,182,600
424-04	Recycle STC Vaporizer	1,840,700
429-06	Recycle STC Superheater	3,098,700
429-08	Recycle H_2 Superheater	1,575,100
434-02	Stripper Condenser	170,000
432-02	Stripper Reboiler	1,095,300
434-06	TCS Column Reboiler	8,536,000
434-08	TCS Column Condenser	8,623,500
434-10	DCS Column Reboiler	5,320,000
434-12	TCS Cooler	1,230,000
434-14	DCS Column Condenser	4,480,000
434-16	DCS Cooler	220,000
434-18	Silane Column Condenser	174,100
432-08	Silane Column Reboiler	650,600
432-26	Refrigerant Heater	45,000
434-24	STC Cooler	649,800
444-02	Pyrolysis Hydrogen Cooler	237,000
444-04	Shotting Hydrogen Cooler	2,800

4.2 Stream Catalog

The stream catalog contains the flow rates and physical properties of the streams for the 1000 MT/Yr mass balance. The stream numbers correspond to the process flow diagram (Figure 2).

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STREAM NUMBER	100	101	102	104	105	106	107
TEMPERATURE, DEG F.	0.75002E+02	0.75002E+02	0.75002E+02	0.30000E+03	0.15001E+03	0.75002E+02	0.76712E+02
PRESSURE, PSIA	0.15000E+02	0.15000E+02	0.20000E+02	0.51670E+03	0.51670E+03	0.14700E+02	0.51670E+03
LIQUID FRACTION	0.9	0.0	0.10000E+01	0.0	0.0	0.0	0.0
VISCOSITY, CENTIPOISE	0.18187E-01	0.0	0.45648E+00	0.11581E-01	0.99447E-02	0.18385E-01	0.94041E-02
THER.COND., BTU/HR-FT-F	0.14779E-01	0.16538E+00	0.58240E-01	0.14218E+00	0.11906E+00	0.14773E-01	0.10714E+00
DENSITY, LB/FT**3	0.79688E-01	0.55042E+03	0.84732E+02	0.12566E+00	0.15607E+00	0.73813E-01	0.12265E+01
SURFACE TENSION, DYNES/CM	0.0	0.0	0.18946E+02	0.0	0.0	0.0	0.0
PRANDTL NUMBER	0.71329E+00	0.72516E+00	0.36199E+01	0.68130E+00	0.70038E+00	0.71322E+00	0.71351E+00
DEW PT. / BUBBLE PT.,F	0.56583E+02	0.0	0.15362E+03	0.0	0.0	0.56039E+02	0.0
ENTHALPY, BTU/LB	0.12892E+03	0.57100E+02	-0.10553E+02	0.25866E+04	0.20676E+04	0.12874E+03	0.37006E+03
SPECIFIC HEAT, BTU/LB-F	0.23336E+00	0.92000E-01	0.19092E+00	0.34576E+01	0.34662E+01	0.23700E+00	0.64249E+00
FLOW * ENTHALPY, BTU/HR	0.74927E+06	0.0	-0.30799E+04	0.14900E+04	0.11792E+06	0.74924E+06	0.10457E+06
AVERAGE MOLECULAR WEIGHT	0.28756E+02	0.63540E+02	0.16990E+03	0.20160E+01	0.20160E+01	0.28795E+02	0.98783E+01

COMPOSITION, LB/HR

	100	101	102	104	105	106	107
1 HYDROGEN	0.0	0.0	0.0	0.57605E+00	0.57029E+02	0.0	0.57658E+02
2 NITROGEN	0.43526E+04	0.0	0.0	0.0	0.0	0.43523E+04	0.12439E+00
3 ARGON	0.74068E+02	0.0	0.0	0.0	0.0	0.74068E+02	0.0
4 OXYGEN	0.13337E+04	0.0	0.0	0.0	0.0	0.13337E+04	0.0
5 CARBON MONOXIDE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
6 CARBON DIOXIDE	0.26233E+01	0.0	0.0	0.0	0.0	0.26233E+01	0.0
7 METHANE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
8 HYDROGEN CHLORIDE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
9 SILANE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
10 MONOCHLOROSILANE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
11 DICHLOROSILANE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
12 TRICHLOROSILANE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
13 SILICON TETRACHLORIDE	0.0	0.0	0.29185E+03	0.0	0.0	0.0	0.0
14 CHLORINE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
15 STEAM / WATER	0.56921E+02	0.0	0.0	0.0	0.0	0.56921E+02	0.0
16 SILICON	0.33764E+03	0.0	0.0	0.0	0.0	0.0	0.33736E+03
17 COPPER	0.0	0.71737E+01	0.0	0.0	0.0	0.0	0.71737E+01
18 TOTAL POUNDS	0.61575E+04	0.71737E+01	0.29185E+03	0.57605E+00	0.57029E+02	0.58195E+04	0.40232E+03

STREAM NUMBER	108	109	110	111	112	113	114
TEMPERATURE, DEG F.	0.93200E+03	0.12060E+03	0.37501E+03	0.35600E+03	0.35600E+03	0.35600E+03	0.10000E+03
PRESSURE, PSIA	0.51470E+03	0.20000E+02	0.51370E+03	0.57470E+03	0.51270E+03	0.51270E+03	0.51170E+03
LIQUID FRACTION	0.0	0.10000E+01	0.50000E+00	0.10000E+01	0.10000E+01	0.0	0.50000E+00
VISCOSITY, CENTIPOISE	0.28715E-01	0.36055E+00	0.47107E-01	0.11511E+00	0.12005E+00	0.20711E-01	0.30128E+00
THER.COND., BTU/HR-FT-F	0.33578E-01	0.52498E-01	0.25549E-01	0.38666E-01	0.38605E-01	0.15727E-01	0.56006E-01
DENSITY, LB/FT**3	0.31788E+01	0.82225E+02	0.10701E+02	0.59167E+02	0.61423E+02	0.88547E+01	0.60748E+02
SURFACE TENSION, DYNES/CM	0.0	0.16716E+02	0.69470E-01	0.20666E-01	0.23804E+01	0.0	0.16683E+01
PRANDTL NUMBER	0.40074E+00	0.32013E+01	0.84229E+00	0.20155E+01	0.20408E+01	0.23450E+00	0.20419E+01
DEW PT. / BUBBLE PT.,F	0.0	0.15012E+03	0.37501E+03	0.0	0.0	0.0	0.10000E+03
ENTHALPY, BTU/LB	0.22673E+03	-0.24167E+01	0.80208E+02	0.47986E+02	0.50999E+02	0.10585E+03	0.37597E+02
SPECIFIC HEAT, BTU/LB-F	0.19369E+00	0.19269E+00	0.13912E+00	0.27344E+00	0.26746E+00	0.85248E-01	0.21064E+00
FLOW * ENTHALPY, BTU/HR	0.88335E+07	-0.83200E+05	0.0	0.62767E+07	0.13322E+05	0.85214E+07	0.0
AVERAGE MOLECULAR WEIGHT	0.91298E+02	0.16816E+03	0.13652E+03	0.16016E+03	0.15143E+03	0.11657E+03	0.11797E+03
COMPOSITION, LB/HR							
1 HYDROGEN	0.37417E+03	0.0	0.39574E+03	0.21571E+02	0.33197E-01	0.38505E+03	0.38606E+03
2 NITROGEN	0.27565E+01	0.0	0.30456E+01	0.28932E+00	0.44285E-03	0.29033E+01	0.29170E+01
3 ARGON	0.0	0.0	0.0	0.0	0.0	0.0	0.0
4 OXYGEN	0.0	0.0	0.0	0.0	0.0	0.0	0.0
5 CARBON MONOXIDE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
6 CARBON DIOXIDE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
7 METHANE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
8 HYDROGEN CHLORIDE	0.12673E+02	0.0	0.20046E+02	0.73735E+01	0.81144E-02	0.17435E+02	0.17680E+02
9 SILANE	0.85625E-02	0.11747E-02	0.12030E-01	0.34673E-02	0.24404E-05	0.11256E-01	0.11506E-01
10 MONOCHLOROSILANE	0.13313E+01	0.12274E+00	0.29667E+01	0.16354E+01	0.15861E-02	0.24576E+01	0.25624E+01
11 DICHLOROSILANE	0.15645E+03	0.18995E+01	0.51025E+03	0.35381E+03	0.62030E+00	0.31071E+03	0.32512E+03
12 TRICHLOROSILANE	0.86961E+04	0.13965E+04	0.31894E+05	0.23198E+05	0.44529E+02	0.17581E+05	0.18410E+05
13 SILICON TETRACHLORIDE	0.29724E+05	0.33029E+05	0.13694E+06	0.10722E+06	0.23336E+03	0.62194E+05	0.65208E+05
14 CHLORINE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
15 STEAM / WATER	0.0	0.0	0.0	0.0	0.0	0.0	0.0
16 SILICON	0.16865E+01	0.0	0.17750E+01	0.88455E-01	0.16865E+01	0.0	0.0
17 COPPER	0.71737E+01	0.0	0.71737E+01	0.0	0.71737E+01	0.0	0.0
18 TOTAL POUNDS	0.38976E+05	0.34427E+05	0.16978E+06	0.13080E+06	0.28741E+03	0.80493E+05	0.84352E+05

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1 0 0 0   M T / Y E A R   P H Y S I C A L   P R O P E R T I E S

STREAM NUMBER	115	116	117	118	119	120	121
TEMPERATURE, DEG F.	0.10000E+03	0.10000E+03	0.10000E+03	0.10000E+03	0.10000E+03	0.10000E+03	0.10000E+03
PRESSURE, PSIA	0.51370E+03	0.51370E+03	0.57270E+03	0.51070E+03	0.51070E+03	0.51070E+03	0.52170E+03
LIQUID FRACTION	0.10000E+01	0.10000E+01	0.10000E+01	0.0	0.10000E+01	0.0	0.0
VISCOSITY, CENTIPOISE	0.35294E+00	0.35294E+00	0.35294E+00	0.16082E-01	0.35294E+00	0.16082E-01	0.16187E-01
THER.COND., BTU/HR-FT-F	0.54397E-01	0.54397E-01	0.54397E-01	0.62400E-01	0.54397E-01	0.62400E-01	0.62650E-01
DENSITY, LB/FT**3	0.81787E+02	0.81786E+02	0.81787E+02	0.17303E+01	0.81787E+02	0.17303E+01	0.17673E+01
SURFACE TENSION, DYNES/CM	0.80144E+01	0.80143E+01	0.73134E+01	0.0	0.80513E+01	0.0	0.0
PRANDTL NUMBER	0.31410E+01	0.31408E+01	0.31410E+01	0.25745E+00	0.31406E+01	0.25745E+00	0.25810E+00
DEW PT. / BUBBLE PT.,F	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ENTHALPY, BTU/LB	-0.79192E+01	-0.79192E+01	-0.79193E+01	0.22519E+03	-0.79193E+01	0.22519E+03	0.22520E+03
SPECIFIC HEAT, BTU/LB-F	0.19988E+00	0.19990E+00	0.19986E+00	0.41417E+00	0.19987E+00	0.41417E+00	0.41293E+00
FLOW * ENTHALPY, BTU/HR	-0.30562E+05	-0.32832E+06	-0.30563E+05	0.94590E+06	-0.27587E+06	0.94590E+06	0.94596E+06
AVERAGE MOLECULAR WEIGHT	0.15738E+03	0.15738E+03	0.15738E+03	0.20417E+02	0.15738E+03	0.20417E+02	0.20417E+02

COMPOSITION, LB/HR

	115	116	117	118	119	120	121
1 HYDROGEN	0.10168E+01	0.10922E+02	0.10168E+01	0.36496E+03	0.91778E+01	0.36496E+03	0.36496E+03
2 NITROGEN	0.13711E-01	0.14729E+00	0.13711E-01	0.26321E+01	0.12377E+00	0.26321E+01	0.26321E+01
3 ARGON	0.0	0.0	0.0	0.0	0.0	0.0	0.0
4 OXYGEN	0.0	0.0	0.0	0.0	0.0	0.0	0.0
5 CARBON MONOXIDE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
6 CARBON DIOXIDE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
7 METHANE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
8 HYDROGEN CHLORIDE	0.44413E+00	0.47769E+01	0.44413E+00	0.86558E+01	0.40089E+01	0.86558E+01	0.86558E+01
9 SILANE	0.24992E-03	0.26848E-02	0.24992E-03	0.63157E-02	0.22559E-02	0.63157E-02	0.63157E-02
10 MONOCHLOROSILANE	0.10488E+00	0.11267E+01	0.10488E+00	0.38424E+00	0.94668E+00	0.38424E+00	0.38424E+00
11 DICHLOROSILANE	0.14416E+02	0.15486E+03	0.14416E+02	0.25717E+02	0.13013E+03	0.25717E+02	0.25717E+02
12 TRICHLOROSILANE	0.82994E+03	0.89156E+04	0.82994E+03	0.11740E+04	0.74915E+04	0.11740E+04	0.11740E+04
13 SILICON TETRACHLORIDE	0.30133E+04	0.32371E+05	0.30133E+04	0.26241E+04	0.27199E+05	0.26241E+04	0.26241E+04
14 CHLORINE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
15 STEAM / WATER	0.0	0.0	0.0	0.0	0.0	0.0	0.0
16 SILICON	0.0	0.0	0.0	0.0	0.0	0.0	0.0
17 COPPER	0.0	0.0	0.0	0.0	0.0	0.0	0.0
18 TOTAL POUNDS	0.38593E+04	0.41456E+05	0.38593E+04	0.42005E+04	0.34835E+05	0.42005E+04	0.42005E+04

STREAM NUMBER	122	123	124	125	126	127	128
TEMPERATURE, DEG F.	0.96100E+03	0.96901E+03	0.44299E+03	0.10000E+03	0.10000E+03	0.75002E+02	0.75002E+02
PRESSURE, PSIA	0.51670E+03	0.51670E+03	0.52170E+03	0.95000E+02	0.10000E+03	0.20000E+02	0.52670E+03
LIQUID FRACTION	0.0	0.0	0.0	0.10000E+01	0.10000E+01	0.10000E+01	0.10000E+01
VISCOSITY, CENTIPOISE	0.29062E-01	0.28189E-01	0.27952E-01	0.35294E+00	0.35294E+00	0.44965E+00	0.44965E+00
THER.COND., BTU/HR-FT-F	0.12573E+00	0.12591E-01	0.97127E-02	0.54397E-01	0.54397E-01	0.58110E-01	0.58110E-01
DENSITY, LB/FT**3	0.68420E+00	0.61206E+01	0.21107E+02	0.81787E+02	0.81787E+02	0.84883E+02	0.84883E+02
SURFACE TENSION, DYNES/CM	0.0	0.0	0.0	0.14539E+02	0.14444E+02	0.18960E+02	0.87334E+01
PRANDTL NUMBER	0.25426E+00	0.84860E+00	-0.12224E+01	0.31408E+01	0.31406E+01	0.36020E+01	0.36020E+01
DEW PT. / BUBBLE PT.,F	0.0	0.44229E+03	0.44347E+03	0.0	0.0	0.15015E+03	0.44344E+03
ENTHALPY, BTU/LB	0.59650E+03	0.18607E+03	0.95816E+02	-0.79194E+01	-0.79194E+01	-0.11063E+02	-0.11063E+02
SPECIFIC HEAT, BTU/LB-F	0.45445E+00	0.15658E+00	-0.14024E+00	0.19978E+00	0.19980E+00	0.19242E+00	0.19242E+00
FLOW * ENTHALPY, BTU/HR	0.25056E+07	0.63954E+07	0.32932E+07	-0.27587E+06	-0.27587E+06	-0.38409E+06	-0.38409E+06
AVERAGE MOLECULAR WEIGHT	0.20417E+02	0.16818E+03	0.16818E+03	0.15738E+03	0.15738E+03	0.16818E+03	0.16818E+03

COMPOSITION, LB/HR

	122	123	124	125	126	127	128
1 HYDROGEN	0.36496E+03	0.0	0.0	0.91778E+01	0.91778E+01	0.0	0.0
2 NITROGEN	0.26321E+01	0.0	0.0	0.12377E+00	0.12377E+00	0.0	0.0
3 ARGON	0.0	0.0	0.0	0.0	0.0	0.0	0.0
4 OXYGEN	0.0	0.0	0.0	0.0	0.0	0.0	0.0
5 CARBON MONOXIDE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
6 CARBON DIOXIDE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
7 METHANE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
8 HYDROGEN CHLORIDE	0.86558E+01	0.0	0.0	0.40089E+01	0.40089E+01	0.0	0.0
9 SILANE	0.63157E-02	0.11629E-02	0.11629E-02	0.22559E-02	0.22559E-02	0.11747E-02	0.11747E-02
10 MONOCHLOROSILANE	0.38424E+00	0.12151E+00	0.12151E+00	0.94668E+00	0.94668E+00	0.12274E+00	0.12274E+00
11 DICHLOROSILANE	0.25717E+02	0.18805E+01	0.18805E+01	0.13013E+03	0.13013E+03	0.18995E+01	0.18995E+01
12 TRICHLOROSILANE	0.11740E+04	0.13825E+04	0.13825E+04	0.74915E+04	0.74915E+04	0.13965E+04	0.13965E+04
13 SILICON TETRACHLORIDE	0.26241E+04	0.32986E+05	0.32986E+05	0.27199E+05	0.27199E+05	0.33321E+05	0.33321E+05
14 CHLORINE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
15 STEAM / WATER	0.0	0.0	0.0	0.0	0.0	0.0	0.0
16 SILICON	0.0	0.0	0.0	0.0	0.0	0.0	0.0
17 COPPER	0.0	0.0	0.0	0.0	0.0	0.0	0.0
18 TOTAL POUNDS	0.42005E+04	0.34371E+05	0.34371E+05	0.34835E+05	0.34835E+05	0.34719E+05	0.34719E+05

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1 0 0 0   M T / Y E A R   P H Y S I C A L   P R O P E R T I E S

STREAM NUMBER	129	131	132	137	138	200	201
TEMPERATURE, DEG F.	0.35600E+03	0.77000E+02	0.44299E+03	0.32167E+03	0.33998E+03	0.21700E+03	0.21481E+03
PRESSURE, PSIA	0.51270E+03	0.14960E+02	0.52670E+03	0.51255E+03	0.52670E+03	0.55000E+02	0.69600E+02
LIQUID FRACTION	0.10000E+01	0.0	0.10000E+01	0.50000E+00	0.10000E+01	0.10000E+01	0.10000E+01
VISCOSITY, CENTIPOISE	0.11511E+00	0.0	0.50087E-01	0.48378E-01	0.14221E+00	0.24725E+00	0.22803E+00
THER.COND., BTU/HR-FT-F	0.38666E-01	0.14968E-01	0.38947E-01	0.27253E-01	0.38291E-01	0.43561E-01	0.43923E-01
DENSITY, LB/FT**3	0.59167E+02	0.60968E+02	0.42917E+02	0.92040E+01	0.63910E+02	0.75552E+02	0.75640E+02
SURFACE TENSION, DYNES/CM	0.22662E+01	0.0	0.48850E+00	0.70708E-01	0.30964E+01	0.11429E+02	0.10894E+02
PRANDTL NUMBER	0.20144E+01	0.70090E+00	-0.30932E+00	0.77627E+00	0.22333E+01	0.27559E+01	0.27784E+01
DEW PT. / BUBBLE PT.,F	0.0	-0.32022E+03	0.44344E+03	0.32167E+03	0.44344E+03	0.21868E+03	0.21872E+03
ENTHALPY, BTU/LB	0.47996E+02	0.13255E+03	0.92496E+02	0.76411E+02	0.43138E+02	0.16502E+02	0.14686E+02
SPECIFIC HEAT, BTU/LB-F	0.27235E+00	0.17005E+00	-0.99425E-01	0.17333E+00	0.24858E+00	0.20071E+00	0.22123E+00
FLOW * ENTHALPY, BTU/HR	0.62779E+07	0.0	0.32114E+05	0.0	0.14977E+07	0.56811E+06	0.74277E+06
AVERAGE MOLECULAR WEIGHT	0.16016E+03	0.28090E+02	0.16818E+03	0.11797E+03	0.16818E+03	0.16816E+03	0.15870E+03

COMPOSITION, LB/HR

	129	131	132	137	138	200	201
1 HYDROGEN	0.21571E+02	0.0	0.0	0.38606E+03	0.0	0.0	0.0
2 NITROGEN	0.28932E+00	0.23412E+00	0.0	0.29170E+01	0.0	0.0	0.0
3 ARGON	0.0	0.0	0.0	0.0	0.0	0.0	0.0
4 OXYGEN	0.0	0.0	0.0	0.0	0.0	0.0	0.0
5 CARBON MONOXIDE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
6 CARBON DIOXIDE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
7 METHANE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
8 HYDROGEN CHLORIDE	0.73735E+01	0.0	0.0	0.17880E+02	0.0	0.0	0.0
9 SILANE	0.34673E-02	0.0	0.11747E-04	0.11506E-01	0.11747E-02	0.11747E-02	0.27611E+00
10 MONOCHLOROSILANE	0.16354E+01	0.0	0.12274E-02	0.25624E+01	0.12274E+00	0.12274E+00	0.12740E-01
11 DICHLOROSILANE	0.35381E+03	0.0	0.18995E-01	0.32512E+03	0.18995E+01	0.18995E+01	0.28666E+03
12 TRICHLOROSILANE	0.23198E+05	0.0	0.13965E+02	0.18410E+05	0.13965E+04	0.13965E+04	0.13288E+05
13 SILICON TETRACHLORIDE	0.10722E+06	0.0	0.33321E+03	0.65208E+05	0.33321E+05	0.33029E+05	0.37002E+05
14 CHLORINE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
15 STEAM / WATER	0.0	0.0	0.0	0.0	0.0	0.0	0.0
16 SILICON	0.88455E-01	0.33764E+03	0.0	0.0	0.0	0.0	0.0
17 COPPER	0.0	0.0	0.0	0.0	0.0	0.0	0.0
18 TOTAL POUNDS	0.13080E+06	0.33788E+03	0.34719E+03	0.84352E+05	0.34719E+05	0.34427E+05	0.50578E+05

STREAM NUMBER	202	203	204	205	206	207	208
TEMPERATURE, DEG F.	0.21900E+03	0.13021E+03	-0.25600E+02	-0.25600E+02	0.21900E+03	0.21321E+03	0.21740E+03
PRESSURE, PSIA	0.70000E+02	0.65400E+02	0.64400E+02	0.70000E+02	0.70000E+02	0.54800E+02	0.55000E+02
LIQUID FRACTION	0.0	0.0	0.10000E+01	0.10000E+01	0.10000E+01	0.10000E+01	0.0
VISCOSITY, CENTIPOISE	0.13769E-01	0.12398E-01	0.24373E+00	0.53410E+00	0.22897E+00	0.24720E+00	0.13625E-01
THER.COND., BTU/HR-FT-F	0.57433E-02	0.11402E-01	0.70085E-01	0.70117E-01	0.43613E-01	0.43864E-01	0.54722E-02
DENSITY, LB/FT**3	0.16443E+01	0.87653E+00	0.57652E+02	0.90449E+02	0.75339E+02	0.75049E+02	0.13731E+01
SURFACE TENSION, DYNES/CM	0.0	0.0	0.39868E+01	0.18518E+02	0.10801E+02	0.11553E+02	0.0
PRANDTL NUMBER	0.73104E+00	0.43629E+00	0.20309E+01	0.39768E+01	0.27603E+01	0.27664E+01	0.69409E+00
DEW PT. / BUBBLE PT.,F	0.22097E+03	0.0	0.0	0.0	0.22298E+03	0.21555E+03	0.21816E+03
ENTHALPY, BTU/LB	0.80486E+02	0.79921E+02	-0.42806E+02	-0.52989E+02	0.15895E+02	0.15558E+02	0.81003E+02
SPECIFIC HEAT, BTU/LB-F	0.15164E+00	0.16585E+00	0.23758E+00	0.21592E+00	0.21734E+00	0.20292E+00	0.14614E+00
FLOW * ENTHALPY, BTU/HR	0.12692E+07	0.10691E+06	-0.57259E+05	-0.69260E+05	0.55327E+06	0.25344E+07	0.10410E+08
AVERAGE MOLECULAR WEIGHT	0.15436E+03	0.80850E+02	0.80850E+02	0.11240E+03	0.16074E+03	0.16675E+03	0.16636E+03
COMPOSITION, LB/HR							
1 HYDROGEN	0.0	0.92256E+01	0.92256E+01	0.47743E-01	0.0	0.0	0.0
2 NITROGEN	0.0	0.12513E+00	0.12513E+00	0.13660E-02	0.0	0.0	0.0
3 ARGON	0.0	0.0	0.0	0.0	0.0	0.0	0.0
4 OXYGEN	0.0	0.0	0.0	0.0	0.0	0.0	0.0
5 CARBON MONOXIDE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
6 CARBON DIOXIDE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
7 METHANE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
8 HYDROGEN CHLORIDE	0.0	0.56219E+01	0.56219E+01	0.16132E+01	0.0	0.0	0.0
9 SILANE	0.26420E+00	0.56055E+01	0.56055E+01	0.18971E+01	0.11910E-01	0.0	0.0
10 MONOCHLOROSILANE	0.96931E-02	0.13188E+02	0.13188E+02	0.12244E+02	0.30465E-02	0.0	0.14172E+02
11 DICHLOROSILANE	0.16656E+03	0.73904E+03	0.73904E+03	0.72901E+03	0.12010E+03	0.15394E+02	0.14172E+02
12 TRICHLOROSILANE	0.57994E+04	0.53116E+03	0.53116E+03	0.52069E+03	0.74891E+04	0.12095E+05	0.10746E+05
13 SILICON TETRACHLORIDE	0.98031E+04	0.33616E+02	0.33616E+02	0.33562E+02	0.27199E+05	0.15079E+06	0.11776E+06
14 CHLORINE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
15 STEAM / WATER	0.0	0.0	0.0	0.0	0.0	0.0	0.0
16 SILICON	0.0	0.0	0.0	0.0	0.0	0.0	0.0
17 COPPER	0.0	0.0	0.0	0.0	0.0	0.0	0.0
18 TOTAL POUNDS	0.15769E+05	0.13377E+04	0.13377E+04	0.13071E+04	0.34808E+05	0.16290E+06	0.12852E+06

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STREAM NUMBER	209	210	211	212	213	214	215
TEMPERATURE, DEG F.	0.15960E+03	0.15440E+03	0.15440E+03	0.15800E+03	0.15440E+03	0.15440E+03	0.15800E+03
PRESSURE, PSIA	0.50200E+02	0.49200E+02	0.17070E+03	0.84000E+02	0.55000E+02	0.17070E+03	0.85000E+02
LIQUID FRACTION	0.0	0.10000E+01	0.10000E+01	0.10000E+01	0.10000E+01	0.10000E+01	0.10000E+01
VISCOSITY, CENTIPOISE	0.12565E-01	0.22707E+00	0.22707E+00	0.23110E+00	0.22707E+00	0.22707E+00	0.22906E+00
THER.COND., BTU/HR-FT-F	0.54578E-02	0.47786E-01	0.47786E-01	0.47654E-01	0.47786E-01	0.47786E-01	0.47564E-01
DENSITY, LB/FT**3	0.10826E+01	0.79361E+02	0.79361E+02	0.79299E+02	0.79361E+02	0.79361E+02	0.79358E+02
SURFACE TENSION, DYNES/CM	0.0	0.11958E+02	0.10536E+02	0.11731E+02	0.11887E+02	0.10535E+02	0.11606E+02
PRANDTL NUMBER	0.65977E+00	0.27851E+01	0.27410E+01	0.27920E+01	0.27856E+01	0.27410E+01	0.27074E+01
DEW PT. / BUBBLE PT.,F	0.16006E+03	0.15377E+03	0.25394E+03	0.19677E+03	0.16148E+03	0.25394E+03	0.20179E+03
ENTHALPY, BTU/LB	0.71253E+02	-0.57889E+01	-0.59393E+01	-0.38554E+01	-0.58069E+01	-0.59394E+01	-0.39503E+01
SPECIFIC HEAT, BTU/LB-F	0.13554E+00	0.23669E+00	0.23845E+00	0.23799E+00	0.24233E+00	0.23845E+00	0.23240E+00
FLOW * ENTHALPY, BTU/HR	0.77929E+07	-0.63313E+06	-0.64958E+06	-0.18426E+06	-0.35507E+06	-0.28641E+06	-0.18880E+06
AVERAGE MOLECULAR WEIGHT	0.13252E+03	0.13252E+03	0.13252E+03	0.13589E+03	0.13252E+03	0.13252E+03	0.13589E+03

COMPOSITION, LB/HR

	209	210	211	212	213	214	215
1 HYDROGEN	0.0	0.0	0.0	0.0	0.0	0.0	0.0
2 NITROGEN	0.0	0.0	0.0	0.0	0.0	0.0	0.0
3 ARGON	0.0	0.0	0.0	0.0	0.0	0.0	0.0
4 OXYGEN	0.0	0.0	0.0	0.0	0.0	0.0	0.0
5 CARBON MONOXIDE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
6 CARBON DIOXIDE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
7 METHANE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
8 HYDROGEN CHLORIDE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
9 SILANE	0.70924E+01	0.70924E+01	0.70924E+01	0.31151E+01	0.39652E+01	0.31272E+01	0.0
10 MONOCHLOROSILANE	0.21084E+03	0.21084E+03	0.21084E+03	0.92964E+02	0.11788E+03	0.92964E+02	0.57648E+00
11 DICHLOROSILANE	0.78709E+04	0.78709E+04	0.78709E+04	0.33516E+04	0.44005E+04	0.34705E+04	0.23618E+03
12 TRICHLOROSILANE	0.98964E+05	0.98964E+05	0.98964E+05	0.37496E+05	0.55329E+05	0.43635E+05	0.46471E+05
13 SILICON TETRACHLORIDE	0.23168E+04	0.23168E+04	0.23168E+04	0.68492E+04	0.12952E+04	0.10215E+04	0.10874E+04
14 CHLORINE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
15 STEAM / WATER	0.0	0.0	0.0	0.0	0.0	0.0	0.0
16 SILICON	0.0	0.0	0.0	0.0	0.0	0.0	0.0
17 COPPER	0.0	0.0	0.0	0.0	0.0	0.0	0.0
18 TOTAL POUNDS	0.10937E+06	0.10937E+06	0.10937E+06	0.47792E+05	0.61146E+05	0.48223E+05	0.47795E+05

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STREAM NUMBER	216	217	218	219	220	221	222
TEMPERATURE. DEG F.	0.15800E+03	0.26191E+03	0.26240E+03	0.19643E+03	0.18500E+03	0.18500E+03	0.18500E+03
PRESSURE. PSIA	0.85000E+02	0.17060E+03	0.17075E+03	0.16590E+03	0.17090E+03	0.16575E+03	0.55000E+03
LIQUID FRACTION	0.10000E+01	0.10000E+01	0.0	0.0	0.10000E+01	0.10000E+01	0.10000E+01
VISCOSITY. CENTIPOISE	0.22906E+00	0.14609E+00	0.15142E-01	0.13521E-01	0.14469E+00	0.14469E+00	0.14469E+00
THER.COND.. BTU/HR-FT-F	0.47564E-01	0.43206E-01	0.69450E-02	0.73121E-02	0.42632E-01	0.42632E-01	0.42632E-01
DENSITY. LB/FT**3	0.79358E+02	0.69593E+02	0.36867E+01	0.27837E+01	0.68288E+02	0.68287E+02	0.68288E+02
SURFACE TENSION. DYNES/CM	0.11606E+02	0.62879E+01	0.0	0.0	0.59767E+01	0.60033E+01	0.42572E+01
PRANDTL NUMBER	0.27074E+01	0.20803E+01	0.86012E+00	0.48557E+00	0.29482E+01	0.29469E+01	0.27398E+01
DEW PT. / BUBBLE PT..F	0.20179E+03	0.26207E+03	0.26276E+03	0.19642E+03	0.18167E+03	0.17907E+03	0.29741E+03
ENTHALPY. BTU/LB	-0.39503E+01	0.21038E+02	0.82065E+02	0.73544E+02	-0.82619E+01	-0.82325E+01	-0.95418E+01
SPECIFIC HEAT. BTU/LB-F	0.23240E+00	0.25481E+00	0.20892E+00	0.10055E+00	0.33587E+00	0.30799E+00	0.33371E+00
FLOW * ENTHALPY. BTU/HR	-0.18899E+06	0.28093E+07	0.70311E+07	0.39813E+07	-0.36131E+06	-0.44566E+06	-0.99351E+05
AVERAGE MOLECULAR WEIGHT	0.13589E+03	0.13572E+03	0.13563E+03	0.97760E+02	0.97760E+02	0.97760E+02	0.97759E+02

COMPOSITION. LB/HR

	216	217	218	219	220	221	222
1 HYDROGEN	0.0	0.0	0.0	0.0	0.0	0.0	0.0
2 NITROGEN	0.0	0.0	0.0	0.0	0.0	0.0	0.0
3 ARGON	0.0	0.0	0.0	0.0	0.0	0.0	0.0
4 OXYGEN	0.0	0.0	0.0	0.0	0.0	0.0	0.0
5 CARBON MONOXIDE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
6 CARBON DIOXIDE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
7 METHANE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
8 HYDROGEN CHLORIDE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
9 SILANE	0.0	0.0	0.0	0.77345E+02	0.62473E+02	0.77345E+02	0.14873E+02
10 MONOCHLOROSILANE	0.57702E+00	0.23895E+01	0.18104E+01	0.68310E+04	0.55176E+04	0.68310E+04	0.13138E+04
11 DICHLOROSILANE	0.23642E+03	0.93790E+03	0.70195E+03	0.39742E+05	0.32108E+05	0.39742E+05	0.76441E+04
12 TRICHLOROSILANE	0.46517E+05	0.12992E+06	0.83303E+05	0.74809E+04	0.60419E+04	0.74809E+04	0.14387E+04
13 SILICON TETRACHLORIDE	0.10804E+04	0.26790E+04	0.15903E+04	0.31431E+01	0.25383E+01	0.31431E+01	0.60442E+00
14 CHLORINE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
15 STEAM / WATER	0.0	0.0	0.0	0.0	0.0	0.0	0.0
16 SILICON	0.0	0.0	0.0	0.0	0.0	0.0	0.0
17 COPPER	0.0	0.0	0.0	0.0	0.0	0.0	0.0
18 TOTAL POUNDS	0.47842E+05	0.13354E+06	0.85677E+05	0.54135E+05	0.43732E+05	0.54135E+05	0.10412E+05

1 0 0 0 M T / Y E A R P H Y S I C A L P R O P E R T I E S

STREAM NUMBER	223	224	225	226	227	230	231
TEMPERATURE, DEG F.	0.12200E+03	0.12200E+03	0.26701E+03	0.26701E+03	-0.25708E+02	-0.25708E+02	-0.25708E+02
PRESSURE, PSIA	0.54900E+03	0.54800E+03	0.36000E+03	0.36000E+03	0.35400E+03	0.35510E+03	0.35410E+03
LIQUID FRACTION	0.10000E+01	0.10000E+01	0.10000E+01	0.10000E+01	0.0	0.0	0.10000E+01
VISCOSITY, CENTIPOISE	0.19617E+00	0.17347E+00	0.93603E-01	0.93602E-01	0.10388E-01	0.10396E-01	0.56724E-01
THER.COND., BTU/HR-FT-F	0.50254E-01	0.51042E-01	0.33181E-01	0.33181E-01	0.98706E-02	0.98824E-02	0.46522E-01
DENSITY, LB/FT**3	0.74368E+02	0.70057E+02	0.60673E+02	0.60673E+02	0.35358E+01	0.35542E+01	0.28984E+02
SURFACE TENSION, DYNES/CM	0.59398E+01	0.58102E+01	0.32051E+01	0.32051E+01	0.0	0.0	0.30694E+01
PRANDTL NUMBER	0.28030E+01	0.22748E+01	0.21454E+01	0.21454E+01	-0.17127E+00	-0.17121E+00	0.20564E+01
DEW PT. / BUBBLE PT.,F	0.29720E+03	0.22686E+03	0.26249E+03	0.26249E+03	-0.19856E+02	-0.19643E+02	-0.19837E+02
ENTHALPY, BTU/LB	-0.29358E+02	-0.19583E+02	0.29077E+02	0.29077E+02	0.43875E+02	0.43875E+02	-0.68501E+02
SPECIFIC HEAT, BTU/LB-F	0.29682E+00	0.27670E+00	0.29042E+00	0.29044E+00	0.69719E+00	0.69719E+00	0.69719E+00
FLOW * ENTHALPY, BTU/HR	-0.30568E+06	-0.20392E+06	0.29172E+06	0.29175E+02	0.16664E+05	0.53326E+05	-0.25758E+05
AVERAGE MOLECULAR WEIGHT	0.97759E+02	0.97771E+02	0.10597E+03	0.10597E+03	0.32120E+02	0.32120E+02	0.32120E+02

COMPOSITION, LB/HR

	223	224	225	226	227	230	231
1 HYDROGEN	0.0	0.0	0.0	0.0	0.0	0.0	0.0
2 NITROGEN	0.0	0.0	0.0	0.0	0.0	0.0	0.0
3 ARGON	0.0	0.0	0.0	0.0	0.0	0.0	0.0
4 OXYGEN	0.0	0.0	0.0	0.0	0.0	0.0	0.0
5 CARBON MONOXIDE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
6 CARBON DIOXIDE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
7 METHANE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
8 HYDROGEN CHLORIDE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
9 SILANE	0.14873E+02	0.39157E+03	0.11746E+02	0.11747E-02	0.37982E+03	0.12154E+04	0.37603E+03
10 MONOCHLOROSILANE	0.13138E+04	0.12216E+04	0.12214E+04	0.12216E+00	0.0	0.0	0.0
11 DICHLOROSILANE	0.76441E+04	0.44105E+04	0.44101E+04	0.44105E+00	0.0	0.0	0.0
12 TRICHLOROSILANE	0.14387E+04	0.43222E+04	0.43221E+04	0.43222E+00	0.0	0.0	0.0
13 SILICON TETRACHLORIDE	0.60442E+00	0.67593E+02	0.67586E+02	0.67593E-02	0.0	0.0	0.0
14 CHLORINE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
15 STEAM / WATER	0.0	0.0	0.0	0.0	0.0	0.0	0.0
16 SILICON	0.0	0.0	0.0	0.0	0.0	0.0	0.0
17 COPPER	0.0	0.0	0.0	0.0	0.0	0.0	0.0
18 TOTAL POUNDS	0.10412E+05	0.10413E+05	0.10033E+05	0.10034E+01	0.37982E+03	0.12154E+04	0.37603E+03

STREAM NUMBER	232	233	234	235	236	237	238
TEMPERATURE, DEG F.	-0.25708E+02	-0.25708E+02	0.12600E+03	0.12600E+03	0.26240E+03	0.15800E+03	0.21740E+03
PRESSURE, PSIA	0.36000E+03	0.37410E+03	0.37410E+03	0.25000E+02	0.17075E+03	0.85000E+02	0.55000E+02
LIQUID FRACTION	0.10000E+01	0.0	0.0	0.0	0.10000E+01	0.10000E+01	0.10000E+01
VISCOSITY, CENTIPOISE	0.56724E-01	0.10560E-01	0.12786E-01	0.12223E-01	0.14600E+00	0.22906E+00	0.24705E+00
THER.COND., BTU/HR-FT-F	0.46522E-01	0.10101E-01	0.13768E-01	0.12582E-01	0.43296E-01	0.47564E-01	0.43532E-01
DENSITY, LB/FT**3	0.28984E+02	0.38935E+01	0.21440E+01	0.12866E+00	0.69561E+02	0.79358E+02	0.75521E+02
SURFACE TENSION, DYNES/CM	0.30507E+01	0.0	0.0	0.0	0.62793E+01	0.11606E+02	0.11413E+02
PRANDTL NUMBER	0.20564E+01	-0.17014E+00	0.69859E+00	0.63388E+00	0.21527E+01	0.27076E+01	0.27530E+01
DEW PT. / BUBBLE PT.,F	-0.18698E+02	-0.16028E+02	-0.16028E+02	-0.14902E+03	0.26240E+03	0.20179E+03	0.21881E+03
ENTHALPY, BTU/LB	-0.68501E+02	0.43875E+02	0.91994E+02	0.10364E+03	0.21186E+02	-0.39503E+01	0.16588E+02
SPECIFIC HEAT, BTU/LB-F	0.69719E+00	0.69719E+00	0.31097E+00	0.26972E+00	0.26389E+00	0.23242E+00	0.20053E+00
FLOW * ENTHALPY, BTU/HR	-0.57240E+05	0.16663E+05	0.34938E+05	0.39363E+05	0.10136E+07	-0.18899E+03	0.57026E+06
AVERAGE MOLECULAR WEIGHT	0.32120E+02	0.32120E+02	0.32120E+02	0.32120E+02	0.13589E+03	0.13589E+03	0.16822E+03

COMPOSITION, LB/HR

	232	233	234	235	236	237	238
1 HYDROGEN	0.0	0.0	0.0	0.0	0.0	0.0	0.0
2 NITROGEN	0.0	0.0	0.0	0.0	0.0	0.0	0.0
3 ARGON	0.0	0.0	0.0	0.0	0.0	0.0	0.0
4 OXYGEN	0.0	0.0	0.0	0.0	0.0	0.0	0.0
5 CARBON MONOXIDE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
6 CARBON DIOXIDE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
7 METHANE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
8 HYDROGEN CHLORIDE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
9 SILANE	0.83560E+03	0.37979E+03	0.37979E+03	0.37979E+03	0.0	0.0	0.0
10 MONOCHLOROSILANE	0.0	0.0	0.0	0.0	0.57702E+00	0.57703E-03	0.0
11 DICHLOROSILANE	0.0	0.0	0.0	0.0	0.23642E+03	0.23642E+00	0.12220E+01
12 TRICHLOROSILANE	0.0	0.0	0.0	0.0	0.46517E+05	0.46517E+02	0.13496E+04
13 SILICON TETRACHLORIDE	0.0	0.0	0.0	0.0	0.10884E+04	0.10884E+01	0.33027E+05
14 CHLORINE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
15 STEAM / WATER	0.0	0.0	0.0	0.0	0.0	0.0	0.0
16 SILICON	0.0	0.0	0.0	0.0	0.0	0.0	0.0
17 COPPER	0.0	0.0	0.0	0.0	0.0	0.0	0.0
18 TOTAL POUNDS	0.83560E+03	0.37979E+03	0.37979E+03	0.37979E+03	0.47842E+05	0.47843E+02	0.34378E+05

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1 0 0 0 M T / Y E A R P H Y S I C A L P R O P E R T I E S

STREAM NUMBER	239	240	242	243	300	301	303
TEMPERATURE, DEG F.	0.26701E+03	-0.25708E+02	0.26701E+03	0.26701E+03	0.77000E+02	0.15621E+04	0.11200E+04
PRESSURE, PSIA	0.36000E+03	0.35410E+03	0.36000E+03	0.36000E+03	0.25000E+02	0.20000E+02	0.20000E+02
LIQUID FRACTION	0.10000E+01	0.0	0.10000E+01	0.0	0.0	0.0	0.0
VISCOSITY, CENTIPOISE	0.93602E-01	0.10388E-01	0.81062E-01	0.15900E-01	0.11295E-01	0.23065E-01	0.0
THER.COND., BTU/HR-FT-F	0.33181E-01	0.98717E-02	0.31891E-01	0.96848E-02	0.10989E-01	0.29363E+00	0.88681E-01
DENSITY, LB/FT**3	0.60673E+02	0.35374E+01	0.56461E+02	0.56608E+01	0.14070E+00	0.14765E-01	0.32308E+01
SURFACE TENSION, DYNES/CM	0.32051E+01	0.0	0.25998E+01	0.0	0.0	0.0	0.0
PRANDTL NUMBER	0.21453E+01	-0.17127E+00	0.19395E+01	0.46843E+00	0.65822E+00	0.66165E+00	0.18661E+00
DEW PT. / BUBBLE PT.,F	0.26249E+03	-0.19836E+02	0.23403E+03	0.25076E+03	-0.14902E+03	0.0	0.0
ENTHALPY, BTU/LB	0.29077E+02	0.43875E+02	0.34391E+02	0.82963E+02	0.90551E+02	0.13196E+04	0.38350E+03
SPECIFIC HEAT, BTU/LB-F	0.29042E+00	0.69719E+00	0.14539E+00	0.11795E+00	0.26474E+00	0.60261E+00	0.17053E+00
FLOW * ENTHALPY, BTU/HR	0.29175E+06	0.16663E+03	0.57249E+06	0.54663E+06	0.34390E+05	0.33343E+06	
AVERAGE MOLECULAR WEIGHT	0.10597E+03	0.32120E+02	0.98871E+02	0.89748E+02	0.32120E+02	0.10711E+02	0.27998E+02

COMPOSITION, LB/HR

	239	240	242	243	300	301	303
1 HYDROGEN	0.0	0.0	0.0	0.0	0.0	0.47438E+02	0.92915E-01
2 NITROGEN	0.0	0.0	0.0	0.0	0.0	0.0	0.0
3 ARGON	0.0	0.0	0.0	0.0	0.0	0.0	0.0
4 OXYGEN	0.0	0.0	0.0	0.0	0.0	0.0	0.0
5 CARBON MONOXIDE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
6 CARBON DIOXIDE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
7 METHANE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
8 HYDROGEN CHLORIDE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
9 SILANE	0.11747E+02	0.37979E+01	0.11775E+03	0.10601E+03	0.37979E+03	0.18990E+01	0.18990E+01
10 MONOCHLOROSILANE	0.12216E+04	0.0	0.31645E+04	0.19429E+04	0.0	0.0	0.0
11 DICHLOROSILANE	0.44105E+04	0.0	0.73971E+04	0.29867E+04	0.0	0.0	0.0
12 TRICHLOROSILANE	0.43222E+04	0.0	0.58865E+04	0.15643E+04	0.0	0.0	0.0
13 SILICON TETRACHLORIDE	0.67593E+02	0.0	0.80573E+02	0.12981E+02	0.0	0.0	0.0
14 CHLORINE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
15 STEAM / WATER	0.0	0.0	0.0	0.0	0.0	0.0	0.0
16 SILICON	0.0	0.0	0.0	0.0	0.0	0.32865E+03	0.29270E+03
17 COPPER	0.0	0.0	0.0	0.0	0.0	0.0	0.0
18 TOTAL POUNDS	0.10034E+05	0.37979E+01	0.16646E+05	0.66129E+04	0.37979E+03	0.37799E+03	0.29469E+03

STREAM NUMBER	307	308	311	312	313	314	2400
TEMPERATURE, DEG F.	0.26000E+04	0.11200E+04	0.15621E+04	0.10000E+03	0.30000E+03	0.77000E+02	0.10000E+03
PRESSURE, PSIA	0.20000E+02	0.20000E+02	0.20000E+02	0.15800E+02	0.55000E+03	0.50000E+02	0.20000E+02
LIQUID FRACTION	0.0	0.0	0.0	0.0	0.0	0.0	0.0
VISCOSITY, CENTIPOISE	0.0	0.0	0.22833E-01	0.94191E-02	0.11580E-01	0.91464E-02	0.18300E-01
THER.COND., BTU/HR-FT-F	0.39094E+00	0.24771E+00	0.29541E+00	0.10954E+00	0.14226E+00	0.10581E+00	0.15507E-01
DENSITY, LB/FT**3	0.14598E+03	0.14598E+03	0.18579E-02	0.52998E-02	0.13361E+00	0.17465E-01	0.93325E-01
SURFACE TENSION, DYNES/CM	0.0	0.0	0.0	0.0	0.0	0.0	0.0
PRANDTL NUMBER	0.74574E+00	0.66508E+00	0.67890E+00	0.71921E+00	0.68085E+00	0.72381E+00	0.69967E+00
DEW PT. / BUBBLE PT.,F	0.0	0.0	0.0	0.0	0.0	0.0	-0.31554E+03
ENTHALPY, BTU/LB	0.78543E+03	0.38989E+03	0.70162E+04	0.18862E+04	0.25873E+04	0.18071E+04	0.13847E+03
SPECIFIC HEAT, BTU/LB-F	0.17000E+00	0.17000E+00	0.36309E+01	0.34575E+01	0.34576E+01	0.34616E+01	0.24402E+00
FLOW * ENTHALPY, BTU/HR	0.0	0.0	0.33217E+06	0.89298E+05	0.14904E+06	0.18540E+05	0.69635E+04
AVERAGE MOLECULAR WEIGHT	0.28090E+02	0.28090E+02	0.20160E+01	0.20160E+01	0.20160E+01	0.20160E+01	0.28016E+02

COMPOSITION, LB/HR

	307	308	311	312	313	314	2400
1 HYDROGEN	0.0	0.0	0.47344E+02	0.47344E+02	0.57605E+02	0.10260E+02	0.0
2 NITROGEN	0.0	0.0	0.0	0.0	0.0	0.0	0.50289E+02
3 ARGON	0.0	0.0	0.0	0.0	0.0	0.0	0.0
4 OXYGEN	0.0	0.0	0.0	0.0	0.0	0.0	0.0
5 CARBON MONOXIDE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
6 CARBON DIOXIDE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
7 METHANE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
8 HYDROGEN CHLORIDE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
9 SILANE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
10 MONOCHLOROSILANE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
11 DICHLOROSILANE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
12 TRICHLOROSILANE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
13 SILICON TETRACHLORIDE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
14 CHLORINE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
15 STEAM / WATER	0.0	0.0	0.0	0.0	0.0	0.0	0.0
16 SILICON	0.29270E+03	0.37753E+02	0.0	0.0	0.0	0.0	0.0
17 COPPER	0.0	0.0	0.0	0.0	0.0	0.0	0.0
18 TOTAL POUNDS	0.29270E+03	0.37753E+02	0.47344E+02	0.47344E+02	0.57605E+02	0.10260E+02	0.50289E+02

1 0 0 0   M T / Y E A R   P H Y S I C A L   P R O P E R T I E S

STREAM NUMBER	2401	2403	2404	2600	2402	2501	1500
TEMPERATURE, DEG F.	0.10000E+03	0.10000E+03	0.10000E+03	0.10000E+03	0.10000E+03	-0.20992E+02	0.77000E+02
PRESSURE, PSIA	0.20000E+02	0.20000E+02	0.20000E+02	0.70000E+02	0.20000E+02	0.90000E+02	0.16000E+03
LIQUID FRACTION	0.0	0.0	0.0	0.10000E+01	0.0	0.0	0.0
VISCOSITY, CENTIPOISE	0.18380E-01	0.18380E-01	0.23489E-01	0.57693E-01	0.18380E-01	0.98652E-02	0.23193E-01
THER.COND., BTU/HR-FT-F	0.15507E-01	0.15507E-01	0.11473E-01	0.45874E-01	0.15507E-01	0.67431E-01	0.10677E-01
DENSITY, LB/FT**3	0.93325E-01	0.93325E-01	0.13082E+00	0.53380E-01	0.93325E-01	0.11864E+00	0.11181E+01
SURFACE TENSION, DYNES/CM	0.0	0.0	0.0	0.82573E-01	0.0	0.0	0.0
PRANDTL NUMBER	0.69967E+00	0.69967E+00	0.64051E+00	0.64577E+00	0.69967E+00	0.41149E+00	0.67124E+00
DEW PT. / BUBBLE PT.,F	-0.31554E+03	-0.31554E+03	0.0	0.0	-0.31554E+03	0.0	-0.24667E+03
ENTHALPY, BTU/LB	0.13847E+03	0.13847E+03	0.71256E+02	0.38810E+02	0.13847E+03	0.47904E+03	0.65841E+02
SPECIFIC HEAT, BTU/LB-F	0.24402E+00	0.24402E+00	0.12933E+00	0.18572E+00	0.24402E+00	0.11627E+01	0.12774E+00
FLOW * ENTHALPY, BTU/HR	0.23212E+04	0.19432E+05	0.89943E+04	0.15733E+05	0.17862E+05	0.14656E+05	0.27982E+04
AVERAGE MOLECULAR WEIGHT	0.28016E+02	0.28016E+02	0.39256E+02	0.64048E+02	0.28016E+02	0.62225E+01	0.39944E+02

COMPOSITION, LB/HR

	2401	2403	2404	2600	2402	2501	1500
1 HYDROGEN	0.0	0.0	0.92915E-01	0.33197E-01	0.0	0.91778E+01	0.0
2 NITROGEN	0.16763E+02	0.14033E+03	0.0	0.12900E+03	0.12900E+03	0.12377E+00	0.0
3 ARGON	0.0	0.0	0.12423E+03	0.0	0.0	0.0	0.42500E+02
4 OXYGEN	0.0	0.0	0.0	0.0	0.0	0.0	0.0
5 CARBON MONOXIDE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
6 CARBON DIOXIDE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
7 METHANE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
8 HYDROGEN CHLORIDE	0.0	0.0	0.0	0.81144E-02	0.0	0.40089E+01	0.0
9 SILANE	0.0	0.0	0.18990E+01	0.24404E-05	0.0	0.37885E+01	0.0
10 MONOCHLOROSILANE	0.0	0.0	0.0	0.15861E-02	0.0	0.94369E+00	0.0
11 DICHLOROSILANE	0.0	0.0	0.0	0.62030E+00	0.0	0.10026E+02	0.0
12 TRICHLOROSILANE	0.0	0.0	0.0	0.44529E+02	0.0	0.24722E+02	0.0
13 SILICON TETRACHLORIDE	0.0	0.0	0.0	0.23336E+03	0.0	0.54400E-01	0.0
14 CHLORINE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
15 STEAM / WATER	0.0	0.0	0.0	0.0	0.0	0.0	0.0
16 SILICON	0.0	0.0	0.0	0.16865E+01	0.0	0.0	0.0
17 COPPER	0.0	0.0	0.0	0.0	0.0	0.0	0.0
18 TOTAL POUNDS	0.16763E+02	0.14033E+03	0.12623E+03	0.40923E+03	0.12900E+03	0.30595E+02	0.42500E+02

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STREAM NUMBER	1501	0	0	0	0	0	0
TEMPERATURE, DEG F.	0.77000E+02	0.0	0.0	0.0	0.0	0.0	0.0
PRESSURE, PSIA	0.16000E+03	0.0	0.0	0.0	0.0	0.0	0.0
LIQUID FRACTION	0.0	0.0	0.0	0.0	0.0	0.0	0.0
VISCOSITY, CENTIPOISE	0.23193E-01	0.0	0.0	0.0	0.0	0.0	0.0
THER.COND., BTU/HR-FT-F	0.10677E-01	0.0	0.0	0.0	0.0	0.0	0.0
DENSITY, LB/FT**3	0.11161E+01	0.0	0.0	0.0	0.0	0.0	0.0
SURFACE TENSION, DYNES/CM	0.0	0.0	0.0	0.0	0.0	0.0	0.0
PRANDTL NUMBER	0.67124E+00	0.0	0.0	0.0	0.0	0.0	0.0
DEW PT. / BUBBLE PT.,F	-0.24667E+03	0.0	0.0	0.0	0.0	0.0	0.0
ENTHALPY, BTU/LB	0.65841E+02	0.0	0.0	0.0	0.0	0.0	0.0
SPECIFIC HEAT, BTU/LB-F	0.12774E+00	0.0	0.0	0.0	0.0	0.0	0.0
FLOW * ENTHALPY, BTU/HR	0.53814E+04	0.0	0.0	0.0	0.0	0.0	0.0
AVERAGE MOLECULAR WEIGHT	0.39944E+02	0.0	0.0	0.0	0.0	0.0	0.0

COMPOSITION, LB/HR

1 HYDROGEN	0.0	0.0	0.0	0.0	0.0	0.0	0.0
2 NITROGEN	0.0	0.0	0.0	0.0	0.0	0.0	0.0
3 ARGON	0.81733E+02	0.0	0.0	0.0	0.0	0.0	0.0
4 OXYGEN	0.0	0.0	0.0	0.0	0.0	0.0	0.0
5 CARBON MONOXIDE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
6 CARBON DIOXIDE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
7 METHANE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
8 HYDROGEN CHLORIDE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
9 SILANE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
10 MONOCHLOROSILANE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
11 DICHLOROSILANE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
12 TRICHLOROSILANE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
13 SILICON TETRACHLORIDE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
14 CHLORINE	0.0	0.0	0.0	0.0	0.0	0.0	0.0
15 STEAM / WATER	0.0	0.0	0.0	0.0	0.0	0.0	0.0
16 SILICON	0.0	0.0	0.0	0.0	0.0	0.0	0.0
17 COPPER	0.0	0.0	0.0	0.0	0.0	0.0	0.0
18 TOTAL POUNDS	0.81733E+02	0.0	0.0	0.0	0.0	0.0	0.0

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FENCE · 570'

ROADWAY

STC / TCS TANKS

H₂ TANKS

COOLING TOWER

STOR. PAD

MAINTENANCE SHOP

OFFICES

ACID TANK

N₂ TANK

LUNCH & LOCKER ROOM

SWITCH GEAR BLDG.

LAB

PARKING

PROCESS HEATERS

PUMPS & COMPRESSORS

REFRIGERATION PACKAGE

PYROLYSIS & CONSOLIDATION

WASTE TREATMENT AREA

GANTRY AREA (HYDROGENATION DISTILLATION)

SILANE TANKS

ROADWAY

FENCE

FENCE

FENCE

400'

FENCE

570' x 400' = 228 000 FT² = 5.2 ACRES INSIDE FENCE

-42

5.0 1000 MTY
OVERALL PLANT LAYOUT

6.0 PROCESS EQUIPMENT

6.1 Equipment List

The equipment list has been broken down by equipment type: vessels, columns, compressors, etc. The number system used is intended to fit in as well as possible with the Linde standard sub-accounts. The following conventions have been adopted for numbering:

- 1st digit is always No. 4 for process equipment

- 2nd digit signifies the processing area of the plant according to the schedule of 1 - raw materials; 2 - hydrogenation; 3 - distillation/re-distribution; 4 - pyrolysis/consolidation; 5 - waste treatment; 6- yard and utilities.

- 3rd digit signifies the type of equipment according to the schedule of 1 - tanks and bins; 2 - columns; 3 - compressors and blowers; 4 - heat exchangers; 5 - reactors; 6 - pumps; 7 - filters; 8 - solids handling; 9 - miscellaneous.

- Following two digits signify individual equipment piece in no specific order. This is an open list. Even numbers are used for main operating units, and odd numbers for spares of backup units.

Several equipment items which are not considered as "stand alone" items have been consolidated with the associated major equipment.

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TANKS AND BINS

1.	411-02	MG Silicon Storage Bin
2.	411-06	Liquid Hydrogen Tank
3.	421-02	MG Silicon Lock Hopper
4.	421-04	Waste Settler Tank
5.	421-06	Waste Chlorides Tank
6.	421-08	Quench Condenser Receiver
7.	421-10	Crude TCS Storage Tank
8.	421-12	STC Storage Tank
9.	421-14	Recycle Hydrogen Receiver
10.	421-18	MG Silicon/Catalyst Blender
11.	431-04	Silane Storage Tank #1 & Coil
12.	431-06	Silane Storage Tank #2 & Coil
13.	431-08	Silane Storage Tank #3 & Coil
14.	431-10	Silane Storage Tank #4 & Coil
15.	441-04	Silicon Powder Hopper
16.	441-06	Pyrolysis Hydrogen Receiver
17.	441-08	Silicon Shot Bin
18.	451-04	Liquid Burner Surge Tank
19.	451-06	Pressure Relief Catch Tank
20.	451-08	Silica Dust Bin
21.	451-10	Waste Silica Neutralizer
22.	451-12	Strong Muriatic Pump Tank
23.	451-14	Weak Muriatic Pump Tank
24.	451-16	Muriatic Acid Storage Tank
25.	451-18	Transportable Silica Bins
26.	461-02	Hot Oil Expansion Tank

COLUMNS

27.	432-02	Stripper Column & Reboiler Coil
28.	432-04	TCS Column
29.	432-06	DCS Column
30.	432-08	Silane Column & Reboiler Coil
31.	452-02	Muriatic Tailing Column

COMPRESSORS

32.	423-02	Recycle Hydrogen Compressor, Main
33.	423-03	Recycle Hydrogen Compressor, Spare
34.	443-02	Pyrolysis Hydrogen Compressor
35.	443-04	Shotting Hydrogen Blower
36.	443-06	Shotting Hydrogen Blower
37.	443-08	Nitrogen Blower
38.	452-02	Agglomerator Blower
39.	453-04	Waste Gas Induction Blower, Main
40.	453-05	Waste Gas Induction Blower, Spare

HEAT EXCHANGERS

41.	424-02	Quench Condenser
42.	424-04	Recycle STC Vaporizer
43.	434-02	Stripper Condenser
44.	434-06	TCS Column Reboiler
45.	434-08	TCS Column Reboiler

HEAT EXCHANGERS (CONT'D)

46.	434-10	DCS Colum Reboiler
47.	434-12	TCS Cooler
48.	434-14	DCS Column Condenser
49.	434-16	DCS Cooler
50.	434-18	Silane Column Condenser
51.	434-24	STC Cooler
52.	434-26	Refrigerant Heater
53.	444-02	Pyrolysis Hydrogen Cooler
54.	444-04	Shotting Hydrogen Cooler
55.	444-06	Shotting Hydrogen Cooler
56.	454-02	Muriatic Acid Cooler
57.	454-04	Silica Agglomerator
58.	464-06	Dry-Out Heater

REACTORS

59.	425-02	Hydrogenation Reactor
60.	435-02	TCS Redistribution Reactor
61.	435-04	DCS Redistribution Reactor
62.	445-02	Pyrolysis Reactor & Hopper

PUMPS

63.	426-02	Quench Contactor Pump, Main
64.	426-03	Quench Contactor Pump, Spar
65.	426-04	Quench Condenser Wash Pump
66.	426-06	Recycle STC Pump, Main
67.	426-07	Recycle STC Pump, Spare
68.	436-02	TCS Distillate Pump, Main
69.	436-03	TCS Distillate Pump, Spare
70.	436-04	DCS Distillate Pump, Main
71.	456-02	Muriatic Acid Pump #1
72.	456-03	Muriatic Acid Pump, Spare
73.	456-04	Muriatic Acid Pump, #2
74.	456-06	Silica Venturi Pump, Main
75.	456-07	Silica Venturi Pump, Spare
76.	456-08	Tailing Column Pump
77.	466-02	Hot Oil Pump, Main
78.	466-04	Cooling Water Pump, Main
79.	466-05	Cooling Water Pump, Spare

FILTERS

80.	417-02	MG Silicon Unloading Filter
81.	427-02	Crude TCS Filter
82.	437-02	Silane Ultra-Filter
83.	447-02	Silicon Shot Filter
84.	447-04	Silicon Shot Filter
85.	457-04	Combustibles Vent Filter
86.	457-06	Silica Bag Filter #1
87.	457-08	Silica Bag Filter

SOLIDS HANDLING EQUIPMENT

88.	428-04	Metallurgical Silicon Conveyor
89.	448-06	Silicon Shot Conveyor
90.	448-08	Silicon Shot Rotary Valves
91.	448-10	Silicon Shot Scale
92.	458-02	Silica Collection Cyclone
93.	458-04	Bin Packer
94.	458-06	Silica Rotary Air Lock

MISCELLANEOUS

95.	429-02	Quench & Solids Removal Contactor
96.	429-04	Gas Superheater Furnace
97.	449-04	Melting-Shotting Assembly
98.	459-02	Silica Venturi
99.	459-04	Muriatic Scrubber
100.	459-06	Weak Muriatic Scrubber
101.	459-08	Waste Gas Burner #1
102.	459-10	Waste Gas Burner #2
103.	459-12	Waste Gas Burner #3
104.	459-14	Waste Liquid Burner
105.	459-16	Liquid Burner Agitator
106.	469-02	Cooling Tower
107.	469-12	Refrigeration System
108.	469-16	Hot Oil Heating Furnace

NOTE: Approximately 160 pages of Process Equipment
Functional Specifications have been deleted -
This is a typical example.

6.2 Process Equipment Functional Specifications | Issue Date: 6/2/80

Rev: _____

LOW COST SILICON SOLAR ARRAY PROJECT

BIN

EQUIPMENT NO. CP 411-02   PROJECT NAME 1000 MT/YR JPL

EQUIPMENT NAME Metallurgical Grade Silicon Storage Bin

NO. REQUIRED 1

TYPE: HORIZONTAL ☐   VERTICAL ☒   OPEN ☐

SUPPORT: CRADLE ☐   STAND ☒   GUSSETED LUGS ☒

HEIGHT 24' Overall   DEPTH/WIDTH 16' x 16'   MATERIAL OF CONSTRUCTION Steel

TEMP: DESIGN Ambient   OPERATING Ambient

PRESS: DESIGN 5" W.C.   OPERATING 2" W.C.

SOLIDS TO BE HANDLED Silicon Powder   BULK DENSITY 70 lb/cu. ft.

ANGLE OF REPOSE Greater than 45°   MATERIAL CHARACTERISTICS abrasive (7.0 MOH)
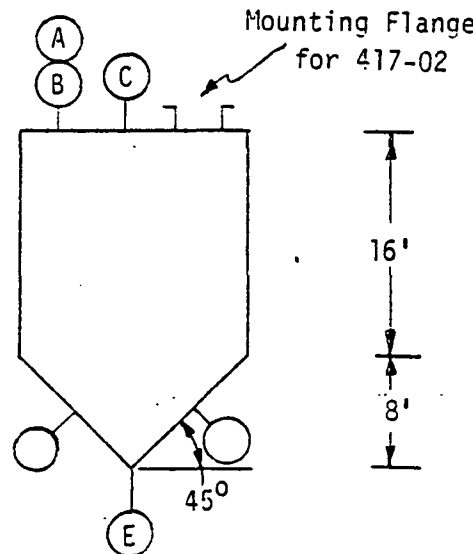
NORMAL WORKING VOLUME 3300 cu ft   INERTING/VENTING

TYPE OF HEADS Flat top, 45° Slant bottom   INTERNAL STRUCTURE none

NOZZLE SCHEDULE				SKETCH
MARK	NO.	SIZE	DUTY	
A	1	½"	Pressure Gauge	
B	1	18"x18"	Hatch	
C	1	6"	Inlet	
E	1	3"	Outlet	

SKETCH:

Mounting Flange for 417-02

16'

8'

45°

Screwed - "A

Flanged - "C, E"

REMARKS